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Imperial Oil

Imperial Oil,
Products and Chemicals Division
Research Department
Sarnia, Ontario, Canada

PROPRIETARY

SARNIA RESEARCH CENTRE - 1993 REVIEW

H. A. Boucher
H. C. Henry
& Research Staff



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INTRODUCTION

1993 was a productive year in the Sarnia Research Centre in terms of technical achievements and associated initiatives aimed at improving the quality of our technical products and our Operations support system.

Health and safety was a continuing high priority area. The monthly publication of "Safety Stride" has continued to focus awareness on safety issues. During 1993, the Health and Safety Committee focused on improvement of several of their processes, including the upgrading of the co-op orientation program and the opening of a new training centre.

Several initiatives were pursued in the Department during 1993 which are expected to have far-reaching consequences on how we carry out our jobs and interact with our customers. These initiatives include Operations Integrity, ISO 9000 quality management, the introduction of Shared Research Planning and Customer Satisfaction workshops and surveys, among others. Because of their importance, special articles describing them are included in this report following the technical sections.

Operations Integrity, or OI, is an operational managing system geared to reduce the potential for incidents that could harm people or the environment and reduce financial and legal risks. The development of a Departmental OI methodology was initiated in 1993, and will continue throughout 1994. Analogously, ISO 9000 International Standards are recognized as sustainable quality frameworks to ensure consistently high quality products. The Engine and Drive Train Lubricants section of the Products Division sought and obtained ISO 9001 certification in 1993. Other sections will be pursuing ISO accreditation during 1994.

Shared Research Planning is a new approach to the planning and funding of Products research work of interest to more than one Exxon region. It replaced the World Mutual Products research planning system in 1993.

Other departmental initiatives included customer satisfaction workshops, which sought to define how we interact with our many customers, and surveys to identify the issues considered to be important by our customers.

The body of this report summarizes the technical achievements made at SRC during 1993, as well as associated engineering progress at Florham Park. Additional details are available in individual memoranda and reports issued during the year. Expenditures for 1993 are shown in the following table. Of the overall SRC effort, 69% was devoted to near to mid-term R&D, 20% to short term research and technical service and 11% to longer range exploratory programs. Imperial Oil Products was again the largest customer, supporting about 50% of our effort. Exxon support amounted to 42%, most of this being directed at the development of Lube Process technology, for which SRC has Exxon-wide responsibility.

1993 PRODUCTS RESEARCH, k\$

	<u>Exploratory</u>	<u>R&D</u>	<u>S/T Research Tech. Service</u>	<u>Total</u>
IMPERIAL OIL				
Products Division				
Lubes and Specialties		4905	1497	6402
Supply		5502	221	5723
Industrial Wholesale		1101	917	2018
Refineries		68	1107	1175
XLT	1297	---	---	1297
Automotive		470	45	515
Site Remediation		196	84	280
Natural Gas Liquids		200	---	200
Branded Associates		59	39	98
Marketing Eng.		---	25	25
Distribution		---	20	20
Total	1297	12501	3955	17753
Resources Division				
Chemicals Division		432	57	489
TOTAL IMPERIAL OIL	1297	13074	4235	18606
EXXON				
WM Lube Process	2750	8202	742	11694
WM Products		571	7	578
WM Analytical		---	52	52
Affiliate Billed		2000	743	2743
TOTAL EXXON	2750	10773	1544	15067
EXTERNAL				
TOTAL RESEARCH	4047	24470	7165	35682

1993 PRODUCTS RESEARCH, k\$

	<u>Exploratory</u>	<u>R&D</u>	<u>S/T Research Tech. Service</u>	<u>Total</u>
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		141	223	364
TOTAL IMPERIAL OIL	1297	13074	4235	18606
EXXON				
WM Lube Process	2750	8202	742	11694
WM Products		571	7	578
WM Analytical		---	52	52
Affiliate Billed		2000	743	2743
TOTAL EXXON	2750	10773	1544	15067
EXTERNAL		623	1386	2009
TOTAL RESEARCH	4047	24470	7165	35682

HIGHLIGHTS

PRODUCTS TECHNOLOGY (see pages 2 - 33 for more details)

Fuel Products

- On-line testing of a GC/MS analyzer at Sarnia Refinery's Mogas Blender has been successful for a period of over six months. Commercialization is targeted for 1Q94.
- A new, rapid mass spectrometric method for the determination of Total Reactive Sulphur species in crudes and refinery streams for better corrosion monitoring has been tested. Field applications are planned in 1994.
- Two potentially lower-cost options relative to ACM treating for the removal of elemental sulphur from pipelined gasolines at the Kamloops terminal have been developed to various stages. A final selection is slated by 1Q94.
- A new corrosion inhibitor package containing MAP-1 has been developed to address silver corrosion in two-cycle snowmobile engines in the Kamloops area. Field testing of a 'gasoline conditioner' is underway.
- Additive cost savings of ~3 M\$ have resulted during 1993 from regional and seasonal optimization of IVC-400 gasoline detergent.
- Ferrocene has been demonstrated to be very effective in reducing the smoke level of higher density domestic fuels. This could enable refineries to divert more cracked stocks into the FFO pool and thereby reduce cetane stress in the diesel pool.
- A potentially cost-effective cetane improver, prepared by modifying tall oil fatty acids, has been identified. Manufacturing and harm test issues will be addressed to establish its potential as an alternative to DII-3 which is currently used.

Asphalts

- Over 1200 tonnes of EB-330 polymer-modified asphalt was used by the Ministry of Transportation of Ontario to pave a 11 km test section of Highway 401. SRC provided manufacturing and construction monitoring support. Pavement condition will be monitored in future years.
- Exxon's proprietary Cosulphonated-SBS binder technology for a higher-performance, lower-cost polymer modified asphalt is being scaled up in preparation for a production test at Strathcona and a pavement trial in Western Canada during 2Q94.

Engine and Drive Train Lubricants

- The EDTL Section obtained ISO 9001 certification in December 1993.
- The PCEO product line was reformulated to the new API 'SH' standard. Commercial production began in August in Sarnia and in October in Strathcona.
- Hydraul Extra, a part synthetic all-season tractor hydraulic fluid, was successfully launched in 3Q93.
- Yamalube 1 Plus was commercialized in early 1993, with unique formulation offering exceptional engine cleanliness and wear protection.
- BLENDWARE program, for blend composition predictions, was developed and validated using 'SH' products manufactured in Sarnia refinery.
- XD-3 Extra core program approvals have just been completed. Commercialization of the Cu passive formulation begins in 1994.
- A viscosity improver study was used to solve the additive drop out problem in some PCEO formulations and to develop a test protocol to identify/overcome this problem in future formulations.

Industrial Oils

- Under the new ASTM viscosity classification system for hydraulic oils, UNIVIS EXTRA is the only truly multigrade premium product on the Canadian market for all-season use.
- Formulas and methods of manufacture were established for all non-IOL metalworking products to be supplied to Exxon U.S.A.
- Development and commercialization of polymer-enriched UNIREX EP greases have been completed. Products included UNIREX EP1, EP1 MOLY, EP2, MOLY H, LOTEMLP EP, LOTEMLP MOLY and UNITOL.
- Development is almost complete on new open gear lubricants DYNAGEAR and DYNAGEAR EXTRA to replace current products which contain trichloroethane carrier.

Lubricant Basestocks

- The lube potential of sour and equity crudes was evaluated for Sarnia and Strathcona. Federated was approved at the 100% level for base stock manufacture at Strathcona.
- Plant tests and SRC results demonstrated a 95 VI, low volatility 100N base oil can be made with normal operating procedures at Sarnia refinery, which can significantly increase light lube production.
- A protocol to manage changes in base stock manufacture was introduced. A crude approval protocol was also developed and approved in principle.

Customer Support

- More than 7700 used oil analyses were carried out, with over 70% being from the Esso Security Plus (ESP) program.

REFINING AND ENVIRONMENTAL (see pages 34 - 47 for more details)

Refining Support

- The Design Specification for the Vancouver Mogas Treater has been issued, capital appropriation has been approved and process start-up is scheduled for 1Q95. The design allows for reduction of elemental sulphur in pipelined Mogas to less than 3 mg/L with no deleterious effects on other Mogas properties.
- Commercial 6" CeraMem ceramic elements were successfully tested at Strathcona for resid ultrafiltration. Flux decline due to foulant dosage was low at a rate of 0 to 0.7% per day. Mechanical sealing issues have been resolved.
- The HCN pervaporation plant test at FOS refinery was successful with more than two months on stream and stable PEI membrane performance throughout the test. A quality control program conducted with Desalination Systems Inc. has shown polymer, membrane and element fabrication to be very reproducible.
- An extensive pilot evaluation of IFP HYC-642 and UOP HC-8 hydrocracking catalysts at commercial operating conditions was successfully completed. Data analysis is in progress.

Environmental Technology and Services

- Bioremediation has the potential to significantly reduce the cost (~\$50/tonne) of clean-up of the former Texaco Refinery site in Montreal. A lab study indicates that bioremediation can achieve the level of hydrocarbon degradation required to meet regulatory requirements, and on-going field trials are confirming the lab results.
- A recycle option was identified for spent polyplant catalyst which will reduce the cost of disposal from \$440 to \$80/tonne, resulting in significant cost savings for both Dartmouth Refinery and Sarnia Chemical Plant.
- A thermal desorption GC method has been developed to analyze BTEX and TPH content of gasoline and mid-distillate contaminated soils in real-time. This method has improved ease of field use, eliminates the need for chlorinated solvents and is expected to reduce soil clean-up volumes.

LUBE PROCESS TECHNOLOGY (see pages 48 - 71 for more details)

- Lube assays were carried out on 23 crudes, including 11 new crudes.
- The predictions of lube yields and qualities by PC LASSY have been improved by using countercurrent rather than batch extraction data, by adding a 100N extraction to the 150 and 600N lab extractions and by using boiling point curves generated by GCD rather than by 15/5 plus Hivac distillation.
- EXXSYN 6 of high quality was produced in each of the three WISR runs in 1993. Data were obtained to support lowering manufacturing cost by operating on high oil content feeds at higher R101 temperature.
- Severe HYDROFINING has been thoroughly validated for potential applications at Port Jerome and Baytown. A severely hydrofined basestock with only 2.7 ppm sulfur easily passed a critical Sequence III E test.
- Characterization of NCLs using microtools developed in the SILQ analytics program, reveals that small fractions of the basestock can impact on its oxidation tendency and its low temperature fluidity.

- An exploratory catalyst comprising a blend of powdered isomerization catalyst (RTF-10) and a microporous zeolite (TON) has shown exceptional selectivity for raffinate wax conversion and VI improvement.
- Outside technology evaluation suggests that Chevron's Isodewaxing Technology is a significant advance over conventional catalytic and solvent dewaxing.
- A joint EE/EEEL/SRC task force identified high temperature sulphur corrosion as the cause of Augusta extract recovery tower fouling that has resulted in frequent plant shutdowns. Implementation of task force recommendations, which include eliminating use of H₂S-containing fuel gas, is planned in 1994.
- Guided by ketone dewaxing correlations, significant dewaxing yield and throughput credits were achieved at Baton Rouge through alteration of pipestill operation to reduce variance of boiling point distributions.
- The maximum yield of low volatility basestocks, which are volatility limited, can be obtained by a proper balancing of distillation and extraction. Studies of yield optimization were carried out for Strathcona, Baton Rouge and Sarnia.
- The technology to increase lube yield by springing oil from the extract solution and recycling the oil to the extractor was successfully tested at Baytown. A \$1 million/year incentive was estimated for using this technology at Baytown.
- An ion exchange bed for removal of acidic components from NMP has operated continuously for more than half of the year and the total acid number is reduced by 90%. Optimization of the regeneration process is ongoing to reduce NMP losses.

OPERATIONS DIVISION (see pages 72 - 76 for more details)

- A Crude Grade Tracker service employing Gas Chromatographic Distillation/ Mass Spectrometry (GCD/MS) to rapidly produce a whole-crude tBP curve is targeted for commercialization at SRC during 1Q94.
- New mass spectrometric methods for sulphur speciation (including Total Reactive Sulfur) of crudes and refinery streams can accurately and quickly quantify these corrosive materials.
- Both gas chromatographic and on-line supercritical fluid techniques for simulated distillation of lubeoils have been extended and improved to permit better control of lube plant operations.

1993 HEALTH AND SAFETY

Health and Safety continued to be a focus area in the Department during 1993, with emphasis on continuous improvement of some of our processes. The co-op orientation training program was upgraded by the addition of a core series of training videos and a new training centre was opened late in the year.

Several pilot projects were also initiated. In the Lube Process Division, a new hazard checklist was developed and implemented, while the Health and Safety expectations of the Products Division were documented in preparation for ISO accreditation. In the Operations Division, two additional loss control programs were established. These programs allowed employees to log deficiencies or concerns (e.g. regarding procedures, equipment) into a computer program which facilitated root cause analyses.

The publication of Safety Stride was continued in 1993 as a medium for communicating safety issues to the Department. A new safety meeting format for the Department was initiated which required attendance of all employees at one of the four division meetings held monthly. This format has resulted in a faster and more consistent response to Health and Safety issues, both from the Committee and from employees.

The latter part of 1993 was a time of change for the Health and Safety Committee as several experienced members stepped down and new members were elected. At year end, the Committee consisted of four elected members, one from each division, and three appointed members. Another committee member, who will be "certified", as mandated by Bill 208, will be added following an election early in 1994.

The Health and Safety Committee was heavily involved in the Department's Operations Integrity initiative during most of 1993. This will continue as a prime focus area for 1994.

PRODUCTS TECHNOLOGY

OVERVIEW

The Products Technology Division was formed in 1992 as part of the Department reorganization. The mission of the Division is to "enhance the success of our customers and partners in achieving industry leading positions by surpassing their expectations for product research results, technical expertise and strategic technology options". Its prime responsibility is the formulation of new and improved products for Imperial Oil, Products Division and other customers.

In 1993, over 45 products, as diverse as gasoline, asphalt, engine and industrial oils were reformulated to meet the increasing demands of the marketplace and to capture cost savings opportunities.

The Division operates under the Internal Market Economy, ensuring that each program area signs a contract with a business sponsor which defines the budget and major deliverables. Within each program area, the work is broken down into projects which are championed by a specific customer in the business unit and guided by planning groups which meet as required. Financial incentives for each project are developed with the customer. An evergreen project planning system is used to prioritize projects and to ensure that only projects with a high pay-off are pursued.

A key goal in 1993 was to shorten the product development time. A four-stage product development process (PDP) has been defined, and the time taken in each stage for a large number of products has been established. Work is now underway to shorten the time in each stage.

The Division played a leadership role in developing a Customer Satisfaction measurement tool, discussed on page 81 of this report. Time for project execution emerged as one of the major determinants of satisfaction for our customers, reinforcing our efforts to improve in that area.

The Division participated in the new Exxon shared planning system, described on page 80 of this report, and was awarded 1994 projects in the Basestocks and Natural Gas Engine Oil areas.

Finally, we were very pleased that the Engine and Drive Train Lubricants section ended the year on a very upbeat note with the award of ISO 9001 certification on December 21, 1993.

The following technology highlights represent activities throughout 1993. They show the milestones achieved, and indicate the status of programs continuing into 1994.

FUEL PRODUCTS

The major IOP business sponsors of Fuel Products research during 1993 were the Supply, Refining and Marketing (Retail Automotive) Departments. Supply's major thrusts were the development and application of state-of-the-art analytical tools for crude assaying and products blending, technologies for treating pipelined products in Western Canada and approaches to relieve current and future diesel cetane stress. Automotive's mogas detergency program was focussed on additive treat cost reduction. NGL supported a small effort to find ways to differentiate propane as an alternative fuel. Major projects are highlighted in the following sections.

1. Crude and Products Quality Monitoring

(IOP - Supply)

Incentives in excess of 14 M\$/yr exist within IOP for rapidly-obtained information on crude and product quality that can be employed in crude purchasing and running decisions, refinery operations and maintenance, and product quality assurance.

1.1 Crude Grade Tracker

GCD/MS (Gas Chromatographic Distillation/Mass Spectrometry) as a tool for crude grade tracking and molecular characterization is being further refined so that the total boiling point (tBP) curve is within statistical agreement with conventional 15/5 Hivac distillations for both fuels and lubes assays (average difference of 1.2 wt% over most of the boiling range). The volume % tBP curve for a slate of over 20 selected crudes, based on the wt% tBP curve and molecular composition, has demonstrated the potential of the method to produce a precise LV% tBP curve. Refinement of it, optimization of the experimental conditions for molecular type information and the development of advanced computer algorithms are underway. A crude grade tracking tBP service is targeted for commercial operation at SRC in 1Q94.

1.2 Reactive Sulphur Typing in Crudes and Refinery streams

The ability to rapidly track corrosive sulphur species in crudes and products is under development to provide to refiners greater flexibility in crude selection and maintenance scheduling around metallurgical decisions. Credits from reduced incident frequency and capital savings are estimated at 7 M\$/yr within IOP. A new, rapid mass spectrometric method has been developed and successfully applied for the determination of total reactive sulphur (TRS) in crude, reduced crude and product streams sampled around the Sarnia Coker. Preliminary results on eleven crude and product samples show excellent correlation between TRS data from SRC and the standard wet chemical methods from Baytown PRD Labs. Analysis time is approximately 10 minutes with the potential for full automation. Further development is planned for the speciation of the sulphur types, especially disulphides and sulphides. This method is planned for future addition to the crude grader tracker service.

1.3 Fuels Fingerprinting

(Can. Petroleum Products Inst.)

Fuels fingerprinting for the Canadian Petroleum Products Institute (CPPI) using the rapid CGSB (Canadian General Standards Board) GC/MS method developed at SRC has been completed for over 3000 samples of refinery rundown streams and finished products (mogas, distillates and solvents) from refiners across the country. The "fingerprints" have been encrypted and added to the master database, which will be distributed by the CPPI to member companies and government agencies. The CGSB rapid GC/MS method will be balloted this year for acceptance and also presented to ASTM as a proposed GC/MS method for the analysis of gasolines and distillates. Other methods presented at ASTM, either by GC or GC/MS, do not cover the range of components in gasolines (400+) nor do they perform the analysis of gasolines and distillates in less than 15 minutes, which is possible with this new method.

Imperial Oil retains intellectual and property rights to the software and database, while the CPPI owns compiled code and encrypted database distribution rights. This will allow IOL to develop its own enhanced versions to support refining, distribution, marketing and quality assurance functions.

1.4 On-Line Product Blending with GC/MS

(IOP - Supply)

Incentives are projected to be >100 k\$/yr per IOL refinery for improved product quality and cost savings for replacing on-line octane comparator engines and other fuels analyzers with GC/MS on-line analyzers. Two such "Megadata Analyzers" have been developed at SRC during 1993 for mogas and diesel blending applications.

a. Sarnia Refinery Mogas Blender Test

The on-line test at Sarnia Refinery mogas blender with a non-ruggedized, laboratory GC/MS instrument has completed 6 months of service and the hardware has proven to be very durable. A ruggedized instrument has been ordered by the refinery for implementation during 1Q94. Modelling of the GC/MS data by Partial Least Squares (PLS) to predict RON, MON and other properties was done with an initial training set of predominantly summer gasolines. Several variations in blend recipes, as well as higher-RVP fall and winter gasolines, are to be included along with "Chemists' Rules" changes to improve predictions. A final model will be ready in early 1994.

b. Sarnia Distillate Blender

Viking Instruments has completed the initial testing of the ruggedized GC/MS analyzer for Sarnia Refinery's distillate blender. The initial factory test and final acceptance in mid-January 1994 will be followed by installation in 1Q94. IOL's initial proprietary software for distillate modelling will be installed on the Viking package. The properties being predicted will include cetane, distillation characteristics, pour and cloud points and viscosity, along with direct measurement of 2+ring aromatics, H₂S and cetane improver concentrations. Strathcona's instrument from Viking is expected to be delivered

during 2Q94 with an initial model tailored for its installation. Model refinements for Sarnia and Strathcona will continue during 1994.

2. Pipelined Products Treating in Western Canada

(IOP - Supply)

2.1 Pipelined Products Treating at Vancouver

A terminalling agreement was signed in 1993 between Imperial Oil Limited (IOL) and Petro-Canada Products (PCP) to ship finished gasoline and diesel from their respective refineries via the Trans Mountain crude pipeline (TMPL) to the future Vancouver Products Terminal. A design specification based on Aqueous Caustic and Mercaptan (ACM) treating was issued in September 1993 for a grassroots, 40 kB/d Mogas Treater to be built at Petro-Canada's Burnaby Terminal to process IOL's and PCP's combined regular and premium gasolines. The DBM targets for the reduction of elemental sulphur from 50 mg/L to less than 3 mg/L with no deleterious impact on other gasoline properties. Intake valve deposit (IVD) tests on a Ford 2.3L engine carried out at AutoResearch Laboratories demonstrated that the ACM-treated gasoline has no detrimental effect on valve cleanliness. The ACM Treater startup date has been tentatively set for January 1995.

Distillates will be treated with IOL's proprietary MAP-1 copper corrosion inhibitor. This cost-effective additive was commercialized in November 1992 at Kamloops and has proven to be more potent than the Reomet 39 previously employed. Jet fuel is also slated for pipeline shipment, followed by fractionation at the Burnaby Terminal to remove elemental sulphur picked up in the crude pipeline. A pipeline test in 2Q93 on jet A-1 shipped via the TMPL from Strathcona to Kamloops indicated that it picked up about 31 mg/L of elemental sulphur and failed JFTOT, Cu and Ag corrosion. The i-95% distilled fraction contained <1 mg/L elemental sulphur, with JFTOT at 260°C and Cu corrosion values improved to 1 ratings. However, Ag corrosion still failed with a 4 rating, apparently due to the presence of H₂S which was formed from unstable sulphur compounds during distillation. Caustic and clay treating could be necessary.

2.2 Pipelined Products Treating at Kamloops

Incentives in excess of 6.5 M\$/year in transportation cost savings over trucking and RMU costs have been identified for gasoline shipped via the TMPL from Strathcona Refinery to Kamloops Terminal. These incentives will be greater because Imperial Oil will supply Petro-Canada in 1994 at Kamloops, which will result in a 60% volume increase. However, gasoline picks up elemental sulphur which could cause premature wear of in-tank electrical fuel pump copper commutators in automobiles and silver bearing corrosion in two-cycle snowmobile engines in the Kamloops area. Several additive and treating technologies are being developed to mitigate these problems.

a. Treating Options for the Kamloops Terminal

The mini-TCMAH technology for rendering elemental sulphur in gasoline harmless was not recommended for Kamloops since intake valve deposit (IVD) with

TCMAH-treated fuel was significantly higher than IVD with conventional fuel. However, two potentially lower cost options to the ACM Treater, MTM (Methanolic/ Tetramethyl ammonium hydroxide / Mercaptan) and MCM (Methanolic/Caustic/ Mercaptan), are at various stages of development. Both options involve the injection into gasoline of a small amount of reagent that contains water and methanol. The methanol acts as a phase transfer agent that accelerates removal of elemental sulphur to target levels of less than 3 mg/L. Focus now has been placed on the MCM option because of its lower operating cost. Process optimization is in progress to reduce the capital investment to less than that for an ACM treater, prior to selection of a single option in 1Q94.

b. Gasoline Conditioner for Two-Cycle Engines

A snowmobile rig test performed by Arctco Inc. has demonstrated that Esso's proprietary MAP-1 additive at 1000 mg/L treat rate restores bearing life in the test to the expected 75-100 hours versus 13 hours using pipelined Kamloops gasoline. Elemental sulphur in gasoline is believed to be causing premature bearing failure by corroding silver-coated connecting rod bearing cages in Arctic Cat, high-performance 700 cc and 900 cc two-stroke engines. A field test on a 900 cc snowmobile currently in progress with MAP-1 treated gasoline has shown no corrosion after 300 miles service. A "Gasoline Conditioner" (17% MAP-1 in Varsol 3139) packaged in 300 ml bottles is being made available to Arctic Cat dealerships for a winter field test prior to full commercialization in 1994.

3. Gasoline Detergency

(IOP - Automotive)

Treat cost reductions of IVC-400, the additive employed for Intake Valve Deposit (IVD) control in all grades of Esso gasolines, have resulted in savings of about 3 M\$ during 1993. At the end of 1992, IVC-400 treats in RUL, MUL and PUL were at 168 ptb (pounds / 1000 barrels). During 1Q93, BMW IVD tests and 2.2L Chrysler PFI (port fuel injector) "keep clean" tests on Sarnia winter production RUL treated with 110 ptb IVC-400 passed Ford's good gasoline requirements by large margins. As of November 1, all regions across Canada except Montreal and the Atlantic were reduced to the 110 ptb level.

During 1993, SRC set up a 2.3L, twin-spark Ford engine to study fuel effects on IVD and to optimize IVC-400 treat rate on a regional and seasonal basis. The results showed that Sarnia summer RUL was about twice as prone to IVD formation as was winter production. For this reason, the IVC-400 treat rate was only reduced to 130 ptb during the summer of 1993 (April to October).

In 3Q93, initial tests on Dartmouth and Petro-Canada (Montreal) summer RULs showed they were more prone to dirty-up intake valves. GC analyses showed that higher levels of diolefins and heavy olefins could be responsible for the increased IVD. Based on these results, Montreal and Atlantic Canada will remain at 130 ptb during the 1993/94 winter until appropriate treat rates are determined.

Recent tests have demonstrated that IVC-400 at 110 ptb can provide port fuel injector cleanup in a Chrysler 2.2L turbo engine. This information will assist Automotive in promoting the unique features of Esso gasolines during the 1994 campaign.

4. New Water Coalescing Filters

(IOP - Supply)

Tests at Sarnia Refinery on new, water coalescing Pall filters installed between the caustic settling drum and the sand filter on the LCN Merox rundown stream, indicate these filters are resistant to caustic. Also, these coalescers have potential as a cost-effective replacement or debottleneck for sand filters used in Merox treating. Parallel testing of effluent streams from the Pall unit and the sand filter for haze, sediment and total alkalinity indicates that the Pall coalescer has consistently out-performed the sand filter. However, the prefilters needed for extended coalescer life plugged rapidly. Prefilters capable of being cleaned by back-washing needed changing on a daily basis, which is not acceptable. Initial results look promising on a new, teflon-coated, disposable filter designed for 3-6 month life. These filters could be lower-cost options to sand filters in ACM and other treating technologies.

5. Nanticoke Mogas Quality

(IOP - Nanticoke Refinery)

Nanticoke refinery gasoline operation routinely requires a final distillation step for the Reformer (CRU) Stabilizer bottoms. During winter operation, the less severe CRU operating conditions often mean that 99.9% of the product is being distilled over-head to remove only 0.1% of less desirable, high endpoint material. The energy costs associated with this process are \$3000/day, thus, significant cost savings can be realized if the tower could be shut down during the winter months. A capillary GC test program identified and quantified the detrimental components in the Rerun Bottoms and enabled comparison of Nanticoke's finished gasolines to those of other IOL and competitive gasolines. As a result, a set of precise quality criteria has been provided to Nanticoke Refinery that enables the decision for rerun tower shutdown or the need for its operation. The option of using the tower to produce valuable specialty solvents is under consideration.

6. Distillate Cetane Stress Relief

(Imperial Oil, Products)

6.1 Cracked Stock Blending into FFO

(IOP - Supply)

Cetane stress at IOP's Eastern refineries can be partly relieved by blending more cracked stocks into the FFO pool, but this is currently limited by a maximum density specification. If higher density fuels are used, they can lead to excessive smoke and carbon build-up on heat exchangers in furnaces. An incentive of >1M\$ has been identified to raise the density spec for FFO from 0.8811 to 0.8923 g/cc at Sarnia and Nanticoke refineries. Testing in the Research burner lab has shown a combustion improver additive, ferrocene, is very effective in reducing the smoke level of higher density FFO. For example, in an Econo-tech high efficiency domestic furnace, 15 ppm of ferrocene in a 0.8923 density fuel gave equivalent performance to current production FFO during both short term, cold start conditions and longer term cyclic operations. Harm testing has shown no impact on other quality features of FFO. Plans are currently underway to run a field test of a higher density FFO blended with ferrocene combustion improver out of the Sarnia Marketing rack.

(Supply)

6.2 Cetane of Hydrogenated Vegetable Oils

The Saskatchewan Research Council has developed a new process for upgrading vegetable oils into possible high cetane blending components for diesel fuel. This uses severe hydrogenation (1400 psi, 440°C) rather than the conventional upgrading process of transesterification with methanol. Samples prepared by CANMET from Canola and Tall oils were tested at SRC for impact on diesel fuel properties. These materials were confirmed to have high cetane numbers (84 for the Canola product and 53 for the Tall oil product), but as expected, they were very waxy due to the formation of wax in the hydrogenation process. As a result, a 5% treat raised the cloud point of diesel fuel by 9-11°C. This limits their usefulness to blending into summer fuels. This data will be used by EM&R, Ottawa for an economic evaluation of the process.

6.3 Cetane Improver from Tall Oil Fatty Acids

(XLT Exploratory)

Tall Oil is an inexpensive (\$0.14/kg) by-product of the pulp mill industry with annual production of 200 MT in Canada and about 900 MT in the U.S. Cetane improvers derived from Tall Oil Fatty Acids (TOFA) have the potential to save IOL ~3-4 M\$/y over DII-3 that is currently used, if the manufacturing cost of the final product can be held below 1\$/kg.

Tall Oil Fatty Acid Nitrate Ester (TOFANE), prepared in collaboration with Tomah Products Div. of Exxon Chemical by nitration of the fatty alcohols, is about half as potent as commercial DII-3 (2-ethylhexyl nitrate). At 0.3 wt% treat rate in diesel fuel, TOFANE did not have adverse effects on accelerated thermal stability, or cloud, pour or flash points, but did slightly deteriorate ASTM colour. Future work will address the commercial manufacturability of TOFANE in two steps by hydrogenation and nitration of TOFA.

6.4 Diesel Ignition Improvement

(XLT Exploratory)

A co-operative exploratory research program with York University investigating methods to improve diesel ignition quality has shown that cetane number of a diesel fuel and its response to cetane improver (DII-3) can be improved by about 3 units by treatment over silica gel. Similar treatment of a diesel fuel derived from Sarnia hydrocrackate (HCIS) distillate (sulphur and nitrogen <2 wppm) did not give any significant cetane number increase or better response to cetane improver. It appears that trace amounts of nitrogen and sulphur compounds can adversely affect ignition quality of a diesel fuel. Identification of these compounds and methods for their removal are being explored.

7. Distillate Cold Flow

(IOP - Supply/Dartmouth)

7.1 Gulfaks Crude Optimization

Gulfaks is an attractive crude for Dartmouth refinery since every cargo saves ~400 k\$ relative to Brent crude. However, the low wax content of Gulfaks can cause response problems for diesel fuels blended with Middle Distillate Flow Improvers (MDFI).

Low Temperature Flow Tests (LTFT) on lab blends indicated that Gulfaks can be blended up to 60% with other crudes and not cause any response problems. Also, above 80%, blended distillates had sufficiently low cloud points to not require any MDFI. Guidelines to avoid the 60-80% range were provided to Dartmouth to help optimize their crude purchases.

7.2 Faster Cold Flow Test Development

The test currently used for quality control of flow improved diesel fuels at Dartmouth is the LTFT. This test requires a slow cool rate of 1°C/hr, which can take up to 16 hr to run. This can lead to product shipment delays and demurrage costs of ~16 k\$/incident. A faster test would reduce these problems. Testing has shown that increasing the LTFT cooling rate to 3°C/hr makes the test more severe, but the change in severity is not significant for fuels currently blended at Dartmouth. Other, less responsive fuels, however, are very sensitive to cooling rate. A wider selection of Dartmouth production fuels will be tested before recommending any change to the test. Also, the faster, automatic Filter Plugging Point test (SFFP) developed in Europe, is being examined as an alternative.

8. Diesel and Jet Quality

(Imperial Oil, Products)

8.1 Diesel Fuel Lubricity

(IOP - Industrial/Wholesale)

With the reduction of the on-highway diesel fuel sulphur level to 0.05% by October 1994, there are concerns that these fuels could have poor lubricity which may lead to high fuel pump and injector wear problems. The SRC ball-on-cylinder lubricity evaluator (BOCLE) was reactivated to measure the lubricity of a series of low sulphur diesel fuels, including exchange products from Petrocan, Shell and Husky and a fuel from Co-op. All fuels tested had acceptable lubricity, giving a wear scar diameter of 0.59-0.65 mm. The Shell Prairie diesel gave the highest wear at 0.65 mm, consistent with its very low sulphur content (<10 ppm). Other tests confirmed effective wear reduction by the lubricity additive DCI-4A. Over the next year, other low sulphur diesel fuels will be tested for lubricity as they appear on the market, to determine if they can cause fuel pump and injector wear problems.

8.2 Jet A-1 Conductivity Depletion

(IOP - Nanticoke Refinery)

Conductivity depletion of Nanticoke Jet A-1 has become more pronounced since switching to Dupont's Stadis 450 conductivity improver additive (following the discontinued production of Shell ASA-3). It is suspected that interaction with polar materials, most likely present in the crude, is causing its depletion. It has been found that alumina treating, following the existing clay treating step, can stabilize conductivity and improve the fuels thermal stability (JFTOT) with reduced clay treating severity, thus extending clay life. A combination clay/alumina treating method is being developed for Nanticoke refinery.

9. Exhaust Emissions: Mogas and Diesel

(Imperial Oil, Products)

9.1 Dartmouth Reformulated Mogas

(IOP - ESC/Dartmouth Refy)

A study was initiated by Dartmouth Refinery and Engineering Services Canada (ESC) to define the investment requirements for making reformulated gasoline at Dartmouth starting in 1997. SRC provided support by applying the EPA Complex model to baseline and candidate Dartmouth gasolines on a planning basis of limiting benzene to 1 vol% and reducing the summer RVP from 10.5 to 9. The results indicate that oxygenated gasoline is not required but MTBE blending would be required when MMT is phased out in 1995. ESC have developed a relatively low cost option for benzene and RVP control utilizing two existing fractionation towers.

9.2 Diesel PAH Emissions

(IOP - Supply/Industry)

A joint industry program to measure the polycyclic aromatic hydrocarbon (PAH) emissions from two late model diesel engines at ORTECH has been completed. This work, sponsored by the CPPI, examined the effects of both diesel fuel quality and engine technology (a DDC Series 60 heavy-duty engine and a Navistar DTA-466 medium-duty engine) on PAH and nitro-PAH emissions. The Navistar engine was tested both with and without an exhaust oxidation catalyst. Results showed that the PAH and nitro-PAH emissions levels were a function of the fuel PAH content rather than its total aromatics content. The majority of diesel fuel aromatics, which contain a single ring, are therefore not implicated. The oxidation catalyst was very effective in reducing PAH emissions, by ~76%, but unexpectedly increased nitro-PAH levels. This was confirmed with two different sizes of catalyst. A final report is currently being prepared for submission to the CPPI.

10. Alternative Fuels - Propane (LPG)

(Natural Gas Liquids)

10.1 Differentiated LPG Fuel

NGL plan future introduction of Essotane, a differentiated LPG fuel. The use of a "flush oil" (0.1 vol% of Iosol 1520) not only solubilizes residues and carries them through the intake manifold into the combustion chamber, but could be capable of delivering some performance enhancing additives into the engine. A mixture of 100 ppm Powershield (lubricity agent) and 100 ppm IVC-400 (detergent), dissolved in 1000 ppm Iosol 1520 and added to the fuel tank, was carried through to the engine, although most of the Powershield was left behind in the evaporator. Other additives have been evaluated, and a fuel formulation will be tested on 5 new vans operated by Robert Q, a fleet operator in London, Ontario.

10.2 NGL Pipelining

Favourable economics exist for NGL's plans to pipeline finished LPG through the IPPL (InterProvincial Pipeline) from Edmonton to Sarnia. A batch of NGL Condensate was monitored for elemental sulphur pickup from Cromer through Superior to Sarnia during

1H93. The product as received was very clean, containing no elemental sulphur and having a 1A copper corrosion rating. However, the 10% point fraction contained a black, pentane-insoluble material which appeared to be a mixture of magnetic material and asphaltenes. A further 30 batches of NGL Condensate were sampled over the following months and the black residue was shown to be an artifact of the interface and hence not a serious problem. It is believed that since LPG will behave similar to NGL Condensate, its pipelining can be accomplished with no detrimental quality impacts.

ASPHALTS

(Imperial Oil, Products)

IOP Marketing Industrial/Wholesale was the largest sponsor of Asphalt research during 1993, the major thrusts being support to Marketing to promote sales of conventional and polymer-modified asphalts across Canada, and the development of new products to position IOP to meet future performance-based specifications. Supply and Refinery sponsored programs addressed the impacts of ongoing sour-up and heavy-up of crude slates.

1. Marketing Technical Support Projects

(IOP - Industrial/Wholesale)

Over forty MTS projects were undertaken in 1993 to support various aspects of the paving and roofing asphalt business that accounted for about 18 M\$/y EBT during 1993. These projects involved asphalt Coatings, Emulsions, Rejuvenating Oils, Tender mixes, Cutbacks, EB formulations, Recycled-polymer for modified asphalts and numerous other formulations. Some of the major projects are highlighted:

1.1 Highway 401 EB-330 Test Section

During May to July 1993, a 3-lane 11 km test section of Hwy 401, east of London near Putnam was paved with ~1200 tonnes of Esso Engineered Bitumen, EB-330. The Ministry of Transportation of Ontario (MTO) paving project involved the evaluation of three products: conventional 85/100 pen grade, Petro-Canada Premium and Esso EB-330. The EB-330 was manufactured at Ashwarren Intnl. in Kingston and delivered to Armbro just in time for paving. In order to meet MTO's stringent specifications, the polymer-asphalt formulation needed to be adjusted due to the low viscosity asphalt feedstock at Ashwarren. All reformulated batches met MTO requirements for asphalt properties. Construction monitoring was performed for the first 100 tonnes of EB-330 placed. It was determined that Armbro employed high quality paving practices which will likely have no long-term negative impact on the performance of EB-330.

The newly-developed Near-Infrared (NIR) technique for polymer and binder content by direct, on-the-road measurements was also evaluated during this project. This method has the potential to replace the time-consuming, laboratory method of solvent extraction/FTIR. A new probe design would be required for better temperature control under on-road conditions. MTO are interested in this development and it could serve as a sales tool for IOL in the future.

1.2 Engineered Bitumen and Premium Asphalt Site Assessments

The monitoring of road performance of Engineered Bitumen (EB) and Premium Asphalt (PA) applications across Canada, and the documentation of conditions during construction which may impact the long-term pavement performance, is an ongoing activity to support IOL Marketing in its efforts to promote these higher margin products. Esso has 37 sections that were paved since 1985, ten of which have been monitored to date to various degrees of detail (Edmonton SH794, Calgary Crowchild Trail, Calgary Brentwood LRT, Craik Sask. PA, Lloydminster Hwy 16, Ontario Hwy. 401, Toronto Eglinton Ave and Hwy 400, Montreal Rue Beaubien-Fibramix, and Cote St. Luc Rue Kildare). The learnings from these and other site assessments are being incorporated into protocols for manufacturing and laying down of future EB and PA test strips. It is intended to incorporate these into a sales tool that will convince future customers to use Esso's EB and PA products.

1.3 Performance-based SHRP Specifications

As a result of the 5-year U.S. Strategic Highway Research Program (SHRP) that concluded in May 1993, a slate of performance-based specifications have been proposed for paving grade asphalt binders. U.S. and Canadian paving authorities are starting to show much interest in the approach that specifies the rutting (high-temperature) and cracking (low-temperature) propensity of asphalt binders on a SHRP 'grid'. In order to position Marketing to field queries regarding IOP asphalts and capture potential sales in the U.S., preliminary samples of all production (IOCO, Strathcona, Dartmouth) and key products (Premium, Polymer-modified EB-330 and EB-352) have been analyzed according to the SHRP Dynamic Mechanical Analysis (DMA) at PRD labs in Linden, NJ. IOP's asphalts, especially those from Cold Lake crude, offer a range of performance. The EB-330 and EB-352 show performance properties superior to a leading competitive product, Styrelf 13. Future developments will be aimed at providing superior performing binders via chemical and polymer modification.

2. Co-sulphonated SBS Modified Asphalt

Exxon proprietary co-sulphonated SBS (Styrene Butadiene Styrene polymer) asphalt binder technology offers the potential of a lower cost, storage-stable binder with rheological properties superior to EB-352 currently marketed in Western Canada. Laboratory tests at SRC with CL 300/400 pen grade asphalt have identified the need for a co-sulphonation temperature >190°C in order to make a storage-stable product. Also, the mixing sequence (3% polymer, followed by sulphuric acid treatment), mixing time and the need for a neutralizing agent (calcium oxide powder) have been established. These findings have been shared with Strathcona Refinery to get agreement for a one-batch test run in mid-1994. A key outstanding issue is the use of a liquid substitute for calcium oxide powder to simplify handling and minimize product contamination. Binder rheology studies at PRD have confirmed the superior properties of co-sulphonated SBS. Hot mix rheological testing is slated for early 1994 at the MSA labs in France.

A meeting of marketing representatives from Exxon USA, Esso France and Imperial Oil resulted in consensus for the 1994 cost-shared research program to be conducted at the four laboratories (PRD, CR, MSA and SRC). Work at SRC will focus on scaleup for a manufacturing trial at Strathcona refinery and the laying of a test pavement in Western Canada.

3. Refining Projects

(Imperial Oil, Products)

3.1 Asphalt Production at Nanticoke

(IOP - Nanticoke Refinery)

The souring up of Nanticoke Refinery's crude slate with a mixture of Husky Synthetic Crude Oil (SCO) and Cold Lake has significant economic credits and provides a potential opportunity to make asphalt for the Ontario market. A mini asphalt assay on 85/100 pen grade and 150/200 pen grade from a crude mix simulating 50/50 Cold Lake Blend/Husky SCO indicates that 'A' grade asphalts can be produced. Further work is on hold since there are no current plans to enter the Ontario market.

3.2 Strathcona RAF

(IOP - Indus/Whsl., Supply, Scona Refy)

In support of Strathcona Refinery's switch to 100% Peace River crude and its potential impact on roofing asphalt flux (RAF) stock, testing was conducted to assess Weather-o-meter life, contact compatibility, control specification properties and stain index, with current 100% Pembina flux as the base case. Statistically, there was no difference in Weather-o-meter results between the two crudes and the customers (BPCO, IKO) should not see any detrimental performance versus current production. Follow-up visits by Marketing and SRC to BCPO and IKO confirmed that appropriate criteria were used to judge the acceptability of alternate fluxes for these customers.

3.3 Asphalt Assays

(IOP - Dartmouth, IOP - Strathcona, IOL Resources)

The selection of appropriate crudes for asphalt production can have a significant effect on refining economics. Three paving asphalt assays were completed in 1993 on Mandji (Gabon crude at Dartmouth), Federated (Strathcona) and Athabasca (for IOL Resources). Mandji has a significant credit for Dartmouth due to its high resid yield, but it cannot be used as a diluent because it deteriorates asphalt properties. Federated crude residue was unable to meet minimum viscosity specifications for any region in Canada. Athabasca residue was also unable to meet Alberta 'A' minimum specifications for 150/200 pen grade and also had a very high insolubles content. The search for ways to modify hard and soft residues continues.

4. Modification of Residues for Asphalt

(IOP - Supply, Indus/Whsl)

4.1 CASH for Premium Asphalt

Catalytic Asphalt Softening by Heating (CASH) is a process that could find future use to produce Premium Asphalt. The process softens hard residues to produce specification

grade asphalt with a higher penetration index. Preferred catalysts are chlorine-based (PVC, chloroform, carbon tetrachloride) and the search for alternative catalysts, in anticipation of chloride-based catalysts not being desirable in the future, has not yielded a successful candidate. This effort will be curtailed in 1994 following the completion of product quality assessments. An economic assessment of the technology is planned.

4.2 Chemically Modified Asphalts

(XLT Exploratory)

Chemically modified asphalt could be an attractive, lower cost alternative to polymer modified asphalt. Acid modification alters molecular interactions in bitumen to increase or decrease its viscosity. A number of candidate acids (stearic, adipic, sulphuric) have been screened with a range of responses. Preliminary tests with sulphuric acid-treated Cold Lake 300/400 pen grade asphalt showed properties superior to EB-352 and EB-330 in terms of SHRP performance criteria for high temperature rutting and low temperature cracking and fatigue. Also, chemically modified asphalt is single phase and storage stable, unlike polymer modified asphalt. Further work is planned to better define this product.

ENGINE AND DRIVE TRAIN LUBRICANTS (EDTL)

The Engine and Drive Train Lubricants section is responsible for technology development and support of the Lube and Specialties business projects. It is responsible for lubricants in four product lines: Passenger Car Engines (PCEO), Heavy Duty Engines (HDEO), Natural Gas Engines (NGEO) and Drive Train (DTL). The section is also responsible for the support of product manufacturing and performance in the field. In the following sections, a summary of the progress of the 1993 projects is presented.

In addition, one of the major objectives of the EDTL section in 1993 was to prepare for ISO 9001-87 accreditation. This effort is also summarized.

1. Heavy Duty Engine Lubricants

1.1 Product Line Development

(Imperial Oil, Products)

1.1.1 Essolube HPD

Reformulation of Essolube HPD, an IOP exclusive super high performance diesel, low ash 10W-40 product, to provide significantly improved upper piston deposit control in emission controlled over-the-road HD diesel engines is continuing. HPD will offer superior high and low temperature viscometrics along with reduced phosphorous content for catalyst compatibility. Product commercialization is scheduled for 2Q94.

Product development has required a combination of bench, engine and field test work. The API CF-4 engine test approval program is in progress and will be completed by the end of 1993. MACK EO-L and GM 6.2L wear test credentials will be established in 1994. Field test evaluation, in Caterpillar 3406C series engines, of four formulations to establish copper passivity and define the preferred formulation, is looking very promising. PC-6 (API 'CG') quality approvals will be initiated following product introduction. It is expected that this new product will also be PC-6 capable.

1.1.2 Essolube XD-3 EXTRA

XD-3 EXTRA development is underway with the goal of eliminating the observed field copper corrosivity of the current commercial multigrade product, while seeking to reduce product cost. Plans for commercialization of XD-3 EXTRA in 1993 have been delayed until mid 1994 by unexpected technical hurdles. Introduction of the new product is now likely to precede the PC-6 (API 'CG') category by only 6 months, so technical targets also include meeting PC-6 requirements.

To meet these needs, a modified Oronite additive system (DI) was selected for the single and multigrade XD-3 EXTRA products. For multigrades, a shear stable PARAMINS VII (non-dispersant OCP) is used. Approval testing is well along with clear passes in the most critical tests.

XD-3 EXTRA will be launched with API CF-4/SH and MACK EO-L credentials for the 15W-40 and 10W-30 grades. The 15W-40 grade will also carry GM 6.2L wear test performance endorsement. SAE 30 and 40 grades are expected to carry API CF/CF-2/SH and MACK EO-L credentials. Both 15W-40 and SAE 30 grades will meet MIL-L-2104F quality. PC-6 (API 'CG') quality approvals will be initiated following product introduction.

1.1.3 PTL Products

PTL product support in 1993 focused on the development of Torq-Guard Supreme + 50, a new 15W-40 engine lubricant for John Deere. Development is expected to be complete by year end 1993, and commercialization is expected 1Q94.

1.2 Field Tests

(Imperial Oil, Products)

The heavy duty engine lubricant field tests fulfill a number of important requirements associated with the development and marketing of Imperial HDEO. These include examining the performance of current IOP HDEO in new engines, obtaining data on competitors' products, and demonstrating field performance of newly developed products.

In 1993, field tests at two sites were concluded (Detroit Diesel Series 60 and CAT 3406C engine types), while three new field tests were designed and established (Detroit Diesel Series 60, CAT 3406C and Cummins N14 engine types) to develop and demonstrate new generation HDEO technologies. These field tests will provide data in all three key Class 8 truck engine types for both competitive and new generation IOP products.

Comparison of current XD-3 EXTRA, Shell Rotella T®, Mobil Delvac 1200 Super® and a 2.0 wt% ash European Esso SHPD product in Detroit Diesel Series 60 engines demonstrated little difference in performance amongst these four oils. All products performed well, but Top Groove Fill (TGF) tended to be high.

Assessment of current XD-3 EXTRA, new generation XD-3 EXTRA technology and experimental low ash 10W-40 products in CAT 3406C engines supported the product line development work discussed in Section 1.1. Oronite XD-3 EXTRA technology delivered good control of wear metals and oil consumption while providing better control of crownland carbon deposits than observed with current XD-3 EXTRA and Shell Rotella T®. Low ash field test work enabled an optimum formulation to be selected for the new generation HPD.

1.3 Next Generation Heavy Duty Lubricant Development

1.3.1 Next Generation Formulation Requirements (IOP Exploratory)

Understanding of the composition of antiwear films is important to the development of lower phosphorus-containing crankcase lubricants. These are needed for the next generation technology to reduce ash content, improve fuel economy and enhance emissions catalyst compatibility.

Many additives are able to reduce wear by forming a protective film on a metal surface. The antiwear function is not attributed to the additives themselves, but rather to their decomposition products, which have in the past proven difficult to identify. By making use of a new spectroscopic technique, antiwear films were determined to be composed of inorganic phosphates, irrespective of the type of phosphorus/sulphur-containing antiwear additive used. This indicates that the different wear control capabilities of various antiwear additives cannot be related to the type of film ultimately formed on the surface, but rather is a function of the rate of film formation and interaction with other additives. In addition, it was shown that the film structure changes with time, and that the presence of other additives, such as detergents and dispersants, results in the formation of thinner films.

In 1994, research will focus on the evaluation of potential antiwear supplements for lower phosphorus, lower ash heavy duty engine lubricants with potentially higher fuel economy for the 1998 low emissions diesel engines.

1.3.2 Next Generation HDEO (Imperial Oil, Products)

Heavy duty engine technology during the 1990s will be dominated by OEMs efforts to meet increasingly stringent government legislated exhaust emission standards. In addition, customers are demanding improved fuel economy, engine performance, durability, and service ease. It is expected that the equipment design changes required to meet these needs will stress lubricants significantly more than in the past.

A lubricant has been developed at SRC to meet the requirements of low heat rejection heavy duty diesel engines. It consists of ester type synthetic basestock, and an IOP proprietary experimental additive package. This lubricant was chosen out of 70 candidates provided by 10 suppliers for the Cummins Engine Company's development of a compact, high power output, high temperature diesel engine for the U.S. Army. It was one of only two oils which completed the full evaluation time in the Cummins L-10 engine test. Because of its very long oil life, it has also been chosen as a field test oil by Cummins to evaluate

extended (100,000 mile) oil drain intervals. Based on these excellent performance results, this lubricant additive chemistry was also formulated using conventional mineral basestock for evaluation as a candidate for the next generation XD-3 EXTRA. The basestock change is expected to affect extreme high temperature performance, especially viscosity increase. However, evaluation of the additive chemistry in conventional and non-conventional basestocks has demonstrated that diesel engine piston deposits are not significantly influenced by such a change. Adjustments to dispersant choice, and potential improvements to fuel economy performance, will be incorporated in 1994.

1.3.3 High Fuel Economy HDEOs

(IOP Exploratory, Government)

Improved fuel economy in heavy duty diesel engines is an emerging performance area, and HD equipment manufacturers have identified the need for high fuel economy lubricants. However, several issues exist: i) most worldwide fuel economy regulations are aimed at gasoline service; ii) there is no widely accepted diesel lubricant fuel economy test procedure; iii) no fuel economy regulations exist for diesel fuel.

IOP and the Canadian and Ontario Governments have signed a joint three year research and development project in new formulation technology for energy efficient heavy duty engine oils, drive train oils and diesel fuels for the heavy duty transportation industry. The project spans research in tribology at the lab level, through development and application in new and existing lab, pilot and field tests.

Installation of a 450 Hp, 12.7 liter Detroit Diesel (1994 version) in the Engine Test Lab was completed, the engine was started up, stabilized and mapped. Reference oils to evaluate the response of the engine to viscosity and friction modification will be used in the development of a standard test procedure early in 1994. The test procedure will then be used to screen new energy efficient lubricants and diesel fuels. As well, a Heavy Duty Valve Train pilot scale test stand was modified to measure friction. Preliminary results on initial tests conducted to determine its capability to screen some viscosity/friction modified engine test oils are being analyzed.

2. Passenger Car Engine Oils

2.1 Product Line Development Support

(Imperial Oil, Products)

Early in 1993, additive dropout problems were reported in packaged PCEO from the field, primarily in the 5W-30 grade. This was traced to the styrene-butadiene VII (Lz 7342) used in IOP's SG formulations, which separated from the finished oil on exposure to moderately low temperatures for extended periods of time. As an interim measure and until formulation to 'SH' was completed, modifications to blending procedures and reformulation using lower saturates basestock components were carried out to reduce the likelihood of the problem recurring.

In 3Q93, Protec Ultra, Protec Extra and Essolube HDX Plus multigrades were reformulated to the new API SH standard. All grades meet SH/CD. 5W-30's and 10W-30's have been qualified against EC-II and ILSAC GF-1 while the 10W-40's meet the EC requirements. For supply security, Sarnia production was commercialized with Ethyl additive chemistry and Paramins viscosity modifier, while Strathcona oils were formulated with Paramins additive technology and VII. Both approval programs were conducted in MXT 5/MCT 10, with base oil interchange testing to support MCT 5-based formulations.

The Ethyl-based HDX Plus formulation has been approved against Ford service fill and Chrysler factory fill specifications. API 10W-30 and 20W-50 formulations with Paramins additive chemistry have been approved for Ford service fill, but a pour depressant modification will be required in the other grades to meet Ford's Scanning Brookfield specification. The Paramins formulations have also been qualified against Toyota 'Genuine Oil' service fill specifications.

2.2 Field Testing

(Imperial Oil, Products)

LXT and MXT 5 basestocks were compared in Essolube XDi formulations of API SG quality in extended drain service (5.2L V-8 Chrysler engines). Both showed equivalent and excellent control of wear, viscosity increase and oil consumption in this service. These data will be used to support the reformulation of this product using MXT 5.

2.3 Next Generation Light Duty Technology

2.3.1 High Fuel Economy Esso Ultra Product

(Imperial Oil, Products)

Major OEMs have identified the need for significantly improved fuel economy, as well as other performance specifications, for the 1996+ model year vehicles. This will require improvement of lubricating oil viscometrics and friction modification. Studies of a series of VII's showed that those which provide the lowest high shear viscosity in the 55-65°C temperature range also show the best fuel economy improvement in the low temperature regime of the Sequence VI fuel economy test. For example, changing the VII used in the Toyota Factory Fill formulation increased the Sequence VI equivalent fuel economy improvement from 2.7% to 3.2%. Evaluation of the impact of the viscosity grade, friction modifier and detergent components are on-going.

A Joint Development Agreement was established with Ford Research in 1993. It is expected that this effort will provide IOP with valuable information on future passenger car engine technology, and prove beneficial to our relationship with this key customer.

2.3.2 Low Temperature Performance

(Imperial Oil, Products)

An industry testing protocol was developed by the ASTM D.02.07C task force studying low temperature cold start and pumpability characteristics of engine oils in modern light duty engines. From extensive OEM surveys, a short list of key engines to be used in the

protocol was developed. IOP, among other companies, is supplying one of the test oils (Essolube HD 30) for this industry test program. In support of this ASTM activity, a joint Exxon affiliate cold start/pumpability program is being carried out at SRC's AWCD facility. Start-up of the experimental program was delayed until late 1993 due to vehicle procurement problems. It is expected that AWCD testing will be completed early in 1994. Results from the industry-wide activity will have direct impact on future revisions of the SAE J-300 viscosity classification specification.

2.4 VI Improver Optimization

(IOP and IOP Exploratory)

Polymeric VI improvers (VIIIs) are key components in many lubricants, including those for engine and drive train applications, and industrial oils. Imperial Oil spends approximately 37 M\$ on VIIIs.

In the first part of this study, the characteristics of 28 different VIIIs were determined (thickening efficiencies and solubility properties in several mineral and synthetic basestocks). From these data, matrices of relative blending efficiencies were generated. Blend studies conducted using non-dispersant VIIIs in API 'SH' quality 5W-30 grade formulations showed that they have a significant impact on the 100N/150N basestock ratios required to meet typical finished oil viscometric targets. Shear stability data were also determined and a ranking of the VIIIs was developed. Dispersant VIIIs were evaluated in the carbon black dispersant test (CBDT), which showed DPMA's and Paramins 'Sledgehammer' VII to have the best performance. Optimum combinations of VII systems have been defined for the next generation Essolube XD-3 0W-30 in terms of shear stability and low temperature fluidity. Results from these and on-going studies will be used to assemble a data base of competitive VII properties and recommendations for future VII improver choice.

The second part of this study is to understand the fundamentals of VII function so that formulations with more cost effective VIIIs can be developed. To this end, viscometric measurements under high and low shear conditions, chemical modification of VIIIs, tests of new polymers, understanding of the synergism of VII mixtures, molecular modeling, and NMR spectroscopy were used. An empirical parameter which describes the combined performance - cost-effectiveness of VIIIs was established, which can be used to rank commercial VIIIs. A third part of this study was to apply collected information to support the light duty product line formulation to API SH. Ternary phase diagrams of the VIIIs, DI additive package, and basestocks were found to be of great value in determining the VII compatibility in engine oil formulations. A solubility test protocol was developed and has been applied to the new 'SH' formulations. It was also determined that the CCS viscosity is significantly affected by the DI package and that more accurate control of the DI addition in commercial blend operations can reduce potential quality give-away, and improve basestock utilization.

protocol was developed. IOP, among other companies, is supplying one of the test oils (Essolube HD 30) for this industry test program. In support of this ASTM activity, a joint Exxon affiliate cold start/pumpability program is being carried out at SRC's AWCD facility. Start-up of the experimental program was delayed until late 1993 due to vehicle procurement problems. It is expected that AWCD testing will be completed early in 1994. Results from the industry-wide activity will have direct impact on future revisions of the SAE J-300 viscosity classification specification.

2.4 VI Improver Optimization

(IOP and IOP Exploratory)

Polymeric VI improvers (VIIIs) are key components in many lubricants, including those for engine and drive train applications, and industrial oils. Imperial Oil spends approximately 37 M\$ on VIIIs.

In the first part of this study, the characteristics of 28 different VIIIs were determined (thickening efficiencies and solubility properties in several mineral and synthetic basestocks). From these data, matrices of relative blending efficiencies were generated. Blend studies conducted using non-dispersant VIIIs in API 'SH' quality 5W-30 grade formulations showed that they have a significant impact on the 100N/150N basestock ratios required to meet typical finished oil viscometric targets. Shear stability data were also determined and a ranking of the VIIIs was developed. Dispersant VIIIs were evaluated in the carbon black dispersant test (CBDT), which showed DPMA's and Paramins 'Sledgehammer' VII to have the best performance. Optimum combinations of VII systems have been defined for the next generation Essolube XD-3 0W-30 in terms of shear stability and low temperature fluidity. Results from these and on-going studies will be used to assemble a data base of competitive VII properties and recommendations for future VII improver choice.

The second part of this study is to understand the fundamentals of VII function so that formulations with more cost effective VIIIs can be developed. To this end, viscometric measurements under high and low shear conditions, chemical modification of VIIIs, tests of new polymers, understanding of the synergism of VII mixtures, molecular modeling, and NMR spectroscopy were used. An empirical parameter which describes the combined performance - cost-effectiveness of VIIIs was established, which can be used to rank commercial VIIIs. A third part of this study was to apply collected information to support the light duty product line formulation to API SH. Ternary phase diagrams of the VIIIs, DI additive package, and basestocks were found to be of great value in determining the VII compatibility in engine oil formulations. A solubility test protocol was developed and has been applied to the new 'SH' formulations. It was also determined that the CCS viscosity is significantly affected by the DI package and that more accurate control of the DI addition in commercial blend operations can reduce potential quality give-away, and improve basestock utilization.

3. Natural Gas Engine Oils

3.1 Products

(Imperial Oil, Products)

3.1.1 Development of Low and Medium Ash Natural Gas Engine Oils

In recent years, more severe operation of natural gas engines has greatly shortened the life of current lubricants. A significant number of customers with standard engine installations are running at higher power levels in order to maximize the efficiency of their operation. Also, new engine designs are being installed as cogeneration units in which engine waste heat is recovered as a useful energy source. As a consequence, engines are running at elevated temperatures, increasing thermal stress on the lubricant, engine deposits and wear.

To meet the greater performance demands, 1993 research efforts were directed at developing cost effective formulations with enhanced resistance to oil oxidation/nitration and engine wear/deposits. Development of an in-house low ash (0.45%) core formulation technology, based on careful examination and optimization of the effects of additive components, is well under way. This technology will also serve as a platform for the development of medium ash (1.0%) lubricants, which will be completed in 1994. Natural gas engine oils having 0.4 - 1.0% ash are applicable to a broad spectrum of 4-cycle engines in the Canadian market. In addition, an experimental 1.0% ash natural gas engine oil, QE-616, was field demonstrated this year. Sites with severe or cogeneration operation report that QE-616 is performing well.

3.1.2 Natural Gas Engine Oil for Sour Gas Operation

Customers running natural gas engines on sour gas (containing elevated levels of H₂S) encountered the unique challenge of controlling engine wear caused by acid build-up in the crankcase. During 1992, some of our customers expressed concerns about high engine wear when running with Essolube GLX, a NGEQ that is designed to operate under conventional conditions. In an effort to supply an existing alternate improved product in a short time frame, highly overbased marine lubricants, Exxmar 12TP30 and 24TP30, were supplied to these sites. Field results show some reduction in engine wear with these products. Continued field testing will be required to verify these preliminary results, and may lead to a rebranding of the product to meet this niche natural gas engine application.

4. Drive Train Lubricants

4.1 Products

(Imperial Oil, Products)

4.1.1 Power Steering Fluids

Sarnia Refinery has been approved by General Motors Saginaw Division as the site for the production of Esso Power Steering Fluid 91423. The number and volume of purchased

components in the formulation, as well as critical control specifications, required development of special blending and handling procedures. The same fluid was also approved by Chrysler's MS-5931 specification, after compatibility tests of the fluid and the Chrysler equipment components were completed.

A next generation power steering fluid candidate has been developed to meet the new General Motors low temperature specification. This fluid must have the same performance properties as the current fluid (provided by 91423), but with improved low temperature properties (the maximum Brookfield viscosity at -40°C has been lowered from 45,000 cP to 20,000 cP). In addition, the IOP-developed fluid has improved compatibility with nitrile seals, better shear stability, better oxidation stability and wear and a higher flash point. Further development is on hold pending GM's response to Imperial's business and technical offering.

4.1.2 Tractor Hydraulic Fluids

Ford New Holland 134D approval for Hydraul 56 using Oronite additive chemistry is underway. Three critical performance parameters have to be met to obtain the approval. (1) Satisfactory wear and operation in a 1000 hour field test. This test was completed showing no abnormal wear or operational problems. (2) The Jenkins test (a severe drive train cycling test designed to simulate 5000 hours field service). This was completed at the New Holland test labs and showed that the transmission was in good condition, but the gear components experienced tooth breakage, scuffing and wear damage. The latter problems do not appear to be oil related. (3) The Killer-Clutch test. Results of the test are expected by the end of 1993.

A part-synthetic all-season tractor hydraulic fluid, Hydraul Extra, has been developed to eliminate the need to change oil in the Spring and Fall, and to give better low temperature performance in the Winter. A key feature of the new fluid is minimum viscosity loss due to in-service shear. Hydraul Extra is offered by Imperial as a "field proven" all-season product, using commercial additive technology, and unique base oil and VI improver combination.

4.1.3 Automatic Transmission Fluids

An automatic transmission fluid formulation has been developed to eliminate the marginal flash point of the current product at Strathcona. The current product is based on Ethyl DI, which uses a low viscosity diluent oil to meet the DEXRON II E Brookfield requirement of 20,000 cP at -40°C, in MCT 5 base stock. As a result, the flash point of this product is at or just above the MERCON limit of 177°C. Low treats of 2 cSt PAO raise the flash point and are cost effective. Recent formulations developed using this approach with a Lubrizol additive package meet all viscometric and seal compatibility targets. The product will be commercialized in early 1994.

(Imperial Oil, Products)

4.2 Field Testing

4.2.1 Tractor Hydraulic Fluids

Hydraul Extra was field tested in two John Deere 648D log skidders in Grande Prairie, Alberta, during the 1992 summer and 1992 / 1993 winter seasons. Skidders are amongst the most severe tractor hydraulic fluid applications, and the test was conducted in summer temperatures $>30^{\circ}\text{C}$ and winter temperatures $<-40^{\circ}\text{C}$. The field test lasted for 2000 hours of operation.

Used oil samples from the Front Differential, Rear Differential, Transmission and Hydraulics were evaluated at regular intervals during the test. Iron and copper levels were insignificant, matching the previously used competitive tractor hydraulic fluid. Hydraul Extra, however, showed significantly better shear stability. A complete inspection of all drivetrain components of one skidder confirmed the excellent performance indicated by the used oil samples. All components were exceptionally clean, and showed no evidence of varnish or sludge deposits. Some transmission components showed evidence of overheating or pitting, however, such observations are normal after such severe service, and are not oil related. The other unit was not inspected in view of the similar used oil results.

The field test demonstrated the all-season capability of Hydraul Extra, and supports its full commercialization. The performance of Hydraul Extra continues to be monitored.

4.2.2 Automotive Gear Oils

Anglamol 99 is used in the premium gear oil GX Extra, which is positioned in the marketplace as a field-proven "problem solver". Changes in the additive manufacturing process, necessitated by environmental regulations, could have unforeseen effects on performance. Two field test sites were chosen for the evaluation of the impact of the new Anglamol product on GX Extra performance. Mack trucks with a GVW of about 375,000 pounds will be used at one site (Quebec) to evaluate the new versions of Esso Gear Oil GX Extra 75W-90 in the manual transmissions, dual differentials and final drive planetary gears. Toro ore haulers will be used at the second site (Manitoba) to evaluate new versions of Esso Gear Oil GX Extra 80W-140 in the front and rear differentials and final drive planetary gears.

5. Two Cycle Oils

5.1 Products

(Imperial Oil, Products)

5.1.1 Commercialization of Yamalube 1 Plus

Yamalube 1 Plus was commercialized at the start of the year. The product has a unique formulation which offers exceptional engine cleanliness and wear protection, while meeting the latest requirements of lube injected engines. It was developed through an extensive program of research and field testing, followed by a full factory approval from Sanshin, Japan. The new product has increased outboard lubricant sales above 1992 levels.

5.1.2 Preparation for TC-W3

The TC-W3 classification was introduced in 1992 by the National Marine Manufacturers' Association, to raise the quality standard for outboard motor oils. This requires products with increased detergency to reduce piston deposits and improve overall engine cleanliness, especially with lower quality fuels. TC-W3 also requires oils with enhanced lubricity, for better protection against piston scuffing and seizure.

Due to technical problems with the approval test methods, and due to the slow recognition of the standard by customers, Imperial Oil elected to delay the introduction of a TC-W3 product until the 1994 boating season. During 1993, research efforts focused on evaluating options which meet the new TC-W3 formulation requirements. A Paramins solvent-containing technology was chosen which will be commercialized as Esso TC-W3 Outboard product in 1994.

5.1.3 Candidate Formulations for Yamaha Snowmobile

At the request of Yamaha, a candidate oil was developed which contains no solvent and offers excellent cleanliness and low temperature fluidity, while providing better wear protection under severe conditions. In addition, lab testing demonstrated that the formulation reduces exhaust smoke by up to 80% relative to conventional oils, a differentiable feature which improves the environmental acceptability of the two-cycle engine. Field testing confirmed the excellent detergency/lubricity performance, but could not demonstrate a meaningful reduction in exhaust smoke, due to complex field testing conditions. No further work is planned at this time pending Yamaha's response.

6. Operations Support

6.1. Additives Management (Imperial Oil, Products)

6.1.1 Additive Specifications

Operations Support (OS) became responsible in 1993 for obtaining detailed additive specifications (grease excepted) from all additive suppliers. To date, over 60% of additives are covered by agreements with the suppliers. Much of the paperwork has been completed on the remainder and the target date for their completion will be 2Q94. Most recently, OS also assumed the responsibility for obtaining endorsements on all non-IOP base oil specifications.

6.1.2 Compatibility Table

The 1993 update of the compatibility table was issued in November 1993. Several new categories were added due to the increase in Sarnia's metal working slate of products. This is now considered a "controlled" document under ISO 9001.

6.1.3 Additives Shelf Life for Blend Plants

Shelf life data was received through Toronto supply from additive suppliers. This information was put into table form and distributed to both Sarnia and Edmonton refineries. Future additive agreements will require this additional information.

6.2 Quality Initiatives

(Imperial Oil, Products)

Several FMEA's, for example, Power Steering Fluid, API SH Passenger Car Engine Oils (both at Edmonton and Sarnia), Yamalube 1 Plus, and in-line blending at Sarnia were carried out. The Initial Sample Inspection Requirement (Ford prerequisite) was included in the product launch. Both Edmonton and Sarnia completed this for 'SH' products.

OS participated in an Exxon task force which investigated statistical methodology for the setting of product specifications. The work of the task force has been completed. Final reports and introduction will take place in 1994.

The bi-annual specialties SQC report containing statistical information relating to all tests performed within Industrial Oils, EDTL and the Customer Support Lab was issued. The annual National QC report has also been issued. EDTL SRC, Edmonton QAL, Sarnia QAL and Sarnia Analytical (part-time) are contributing members. This report compares statistical data produced from identical quality control samples.

6.3 Blendware

(Imperial Oil, Products)

'BLENDWARE' is a mathematical software package designed to improve the Lube Blending Operations capability to accurately predict the component recipe required to blend lube products. In combination with business changes, BLENDWARE is expected to improve blending success, minimize rework and produce more consistent product quality. Initial steps to implement Blendware early in 1993 identified design changes that would greatly enhance the software. These changes have delayed the implementation of BLENDWARE till 1994.

The design improvements included widening the empirical model, a larger database, applying linear regression, incorporating field data, using actual rather than typical additive qualities incorporating an LP, and were completed on a demonstration basis for Sarnia refinery introduction of new API SH quality engine oils. Excel 4.0 was used as the framework for 'BLENDWARE' and an optimization subroutine was added to allow optimal recipe prediction based on product specifications, volume and cost constraints. These changes significantly improved the predictive capability of 'BLENDWARE' in the lab. Validation against plant data over four months confirmed the program to be capable in the blend plant.

A checkpoint review with customers on objectives and revised project costs and schedules, will be held early in 1994 before proceeding with development, refinery tests and implementation of the enhanced 'BLENDWARE'.

7. EDTL ISO 9001-87 Certification

The EDTL section underwent a 2 day formal assessment against the ISO 9001-87 standard on Dec. 20 and 21, 1993, and were recommended for certification.

INDUSTRIAL OILS

1. Circulating, Non-Circulating and Gear Oils (Imperial Oil, Products)

Imperial and competitive VI-improved industrial hydraulic oils were tested for shear stability and low temperature properties in order to categorize them under the new ASTM viscosity classification. Results indicated that UNIVIS N oils are industry leaders in their ability to retain viscosity in service, but have poorer low temperature performance in a few instances. UNIVIS EXTRA was shown to be the only truly multi-grade petroleum-based product on the market for all-season use. This new viscosity classification system is endorsed by hydraulic equipment OEM's, and its adoption will lead to replacement of automotive oils (such as ATF), which have poor shear stability in industrial hydraulic equipment, by industrial products such as UNIVIS N.

TERESSO EP 32 was developed for use in Asea Brown Boveri STAL steam turbines. Recent customer information indicated that anti-wear functionality is needed in these oils, and TERESSO EP 32 meets this requirement, passing the FZG gear test at stage 9. Customers currently using TERESSO 32 were assisted to upgrade by top-treating with anti-wear additive.

TERESSO GT 32 was developed for lubricating General Electric Frame 7 type gas turbine equipment. Laboratory tests showed this product to be among the top competitive performers in high temperature oxidation stability, except for PAO-based synthetics. This product will enable IOP to compete for business in these types of gas turbine installations which are often used in large power co-generation plants, which are increasing in number.

As part of the transfer of products from Commissioners blend plant to Sarnia, AW Oil's 100 and 320, for use in steel rod mills, were reformulated to use existing Sarnia additives while retaining OEM approvals from Morgan. This allowed uninterrupted supply to a key customer, Ivaco. For the same reason, a new grade of gear oil, SPARTAN EP 360, was formulated to replace MM OIL 360 for Stelco.

ECA 4570 was identified as a direct replacement for Lubrizol 5531, the detergent and anti-rust additive in TERESSTIC N paper machine oils, since the latter is no longer available. The reformulated oil met all the technical requirements for TERESSTIC N products, and the change will be invisible to customers, except for a shift in additive metals levels.

Testing is now complete on Hitec 9192 anti-wear industrial hydraulic adpack from Ethyl. It meets all industry and OEM requirements, including formal approvals from Cincinnati-Milacron, Hagglunds-Denison HF0 and Vickers. This adpack provides an alternative, and potentially lower cost source to Lubrizol 5178L adpack, which is used in NUTO H, UNIVIS N and ENTECH hydraulic oils. Testing is in progress to determine whether the new adpack may have some differentiated performance features, such as superior oxidation stability.

SRC's mist oil test rig was utilized to examine the performance of Exxon's reformulated ENMIST oils for mist lubrication systems. They were superior in performance to gear and paper machine oils, and to competitive mist oils, since they had a very favourable ratio of high delivery rate to the lubrication site and very low stray mist production. Key technology advantages include the use of naphthenic base stocks to avoid wax dropout, and poly-isobutylene additive to suppress the formation of microscopic stray mist droplets. These products have been commercialized, particularly in the steel and paper industries, for use across North America.

2. Interfacial Properties of Industrial Oils

(ECI/IOP Shared Research)

Due to foaming of base oils, industrial oils require the use of antifoaming agents. Twenty years ago, silicone-type antifoaming agents were eliminated from the majority of Exxon industrial oils due to their negative effect on air entrainment and were replaced by polyacrylate-type antifoaming agents (PC-1244, Mobilad C-402). However, Exxon affiliates continue to report problems related to both foaming and poor air release properties of industrial oils. This project is intended to provide information that will resolve these formulation problems.

The effect of polyacrylate type antifoaming agent PC-1244 on air entrainment of various base oils was studied at active treat rates of 10 and 40 ppm. 10 ppm PC-1244 was found not to affect air entrainment properties of 150N base oils, but doubled the air release times of 600N base oils from an average of 8 min to 16 min. The results of statistically designed experiments showed that the treat rates of PC-1244 required to prevent foaming in different base oils vary from 8 ppm (active) for high viscosity base oil blends (containing mostly 600N) to 80 ppm (active) for low viscosity blends (containing mostly 150N).

Antifoaming agent PC-1244, sold in the form of a 40 wt% solution, is further diluted to 10 wt% by some Exxon blending plants to increase its effectiveness. Results confirmed that an increase in effectiveness of diluted PC-1244 is observed in Teresso oils when tested at 24°C (Seq. I foam test) and at treat rates below 70 ppm (active). At treat rates above 70 ppm (active), the benefit of using a more diluted solution of PC-1244 was eliminated. Recent research in the area of PC-1244 surface activity indicate its effectiveness is due to its negative spreading coefficient at the oil/air interface of some base oils. Work is in progress to evaluate the effect of temperature on the performance of antifoaming agents and to develop guidelines for antifoaming agent selection.

3. Process Oils, Wax and Metalworking Fluids

(Imperial Oil, Products)

Correlations between wax oil contents as measured by ASTM D721 or D3235 and a UV spectrophotometric technique have been derived for Sarnia 5, 10, 30, and 60 slack waxes. The UV method simplifies the oil content measurement and is insensitive to changes in feed stocks or processing conditions. The Process Oil TPG has recommended that Sarnia Refinery adopt the UV procedure and report oil contents as correlated to ASTM D721. It is also recommended that Strathcona Refinery report wax oil content by ASTM D721. UV correlations would be developed for the latter site if required.

Support was provided to the Sarnia blend plant for the manufacture of Exxon metalworking products. Formulas and methods of manufacture have been determined for all non-IOL products. In the course of this project, an additive concentrate was developed for GM Quench Oils which facilitates the manufacturing process.

A new base oil, XT-50, which is the top pump around from the LVIS unit, and which is collected during the production of MXT distillate, has been established. Compositional data on this stream assisted Exxon Biomedical to establish that this product need not be labeled under WHMIS regulations. This 50N base oil is a water-white fluid with very low odour and a relative high flash point (160°C). The stream will be treated with a pour point depressant to give a pour point below -30°C to facilitate storage in unheated tanks, and also with an antioxidant to improve daylight stability. Fully formulated XT-50 has passed the requirements for FDA CFR 178.3620(c), permitting its use in incidental food contact. The product will be utilized in the following applications: Absorbent Oil, a replacement for currently purchased mineral seal oil, replacement for white oil in a paper manufacturing process, EDM fluid, textile fluid, and aluminum rolling oil.

4. Rust-Ban Product Line Reformulation

(Imperial Oil, Products)

An updated Rust-Ban product line is being developed that better meets customer requirements at reduced product costs. Initial work identified opportunities to change the product line by reducing the overall number of products from 6 to 4, which involves development of two new product types and reformulation of two existing products. These opportunities were identified by evaluating the current products using existing and new test methods, visiting customers and benchmarking against competition. The new product lineup consists of a solvent cutback, an oil base, a heavy duty and a water based product; the latter is especially attractive in light of current environmental concerns. The work in 1993 identified the most promising candidate formulations. Project completion in 1994 will involve field testing, establishing application rates, removal methods, and length of service for all products.

5. Grease

(Imperial Oil, Products)

Development and commercialization of polymer enriched UNIREX EP greases was completed in 1993. Products reformulated included UNIREX EP1, EP1 MOLY, EP2, MOLY H, LOTEMP EP, LOTEMP MOLY, and UNITOL. The new products provide

significantly improved water resistance and adhesion. The new low temperature grease formulations will also greatly reduce supply and customer concerns about oil bleeding from tubes and the resulting package staining.

Reformulation of open gear products containing trichloroethane carrier solvent, the use of which is restricted under the terms of the Montreal protocol, was also largely completed in 1993. The existing products, ESSO 40/40 GREASE and ESSO MILL GREASE, will soon be replaced by DYNAGEAR and DYNAGEAR EXTRA in both Canada and the U.S. These products, which have been formulated without the use of carrier solvents of any kind, are currently undergoing final customer evaluations leading to full commercialization in early 1994. A solvent-free version of ESSO WIRE ROPE DRESSING is also in development, with field testing underway at INCO in Sudbury scheduled to be completed 1Q94. Finally, the current slate of 10 asphaltic Gear Cover Fluids, many containing trichloroethane, will be replaced in 1Q94 by four new products containing a hydrocarbon carrier solvent.

Research also supported the relocation of greases manufactured at the Commissioners Street Blending and Grease Plant, which was closed in mid-1993, to other suppliers. Products affected included NEBULA EP2 steel mill grease, DDR GREASE, a number of GEAR COVER FLUIDS, GALENA TRAMO 3735, GALENA MOLY EP and EP0, LUBREX, LONAX, and FILMO A. A number of these transfers required formulation adjustments and adaptation of methods of manufacture to different equipment configurations.

6. Rail and Marine Oils

(Imperial Oil, Products)

During the year, minor reformulations were made to accommodate VII changes in Galena 10RD20W40 and 17RD20W40, and in both additive and base oils in Galena 17RD40 to meet a CN request for product consistency.

Minor changes were also made to the entire EXXMAR line. In Sarnia, this involved substituting a single adpack for several additives, to improve blending capability. In Strathcona, the existing calcium sulphonate for the EXXMAR line was replaced by a new additive and a demulsifier was included in the package. Specifications for all EXXMAR products were made consistent with JY standards. Two new products were formulated for Strathcona: EXXMAR 30TP30 was added for export to EUSA and is aimed at the Pacific market, and IOLUBE MDY40 will supply Canadian government requirements in the N.W.T.

7. Used Oil Processing

(Imperial Oil, Products)

A 'closed loop' strategy has been developed by the Lubricants and Specialties (L&S) Division of IOP. One of the opportunities for this strategy is processing of used oil to create useful refinery feedstocks. In that regard, SRC has supported two projects in 1993. One is implementation of the Shurtleff process in Edmonton and the other is potential application of the Enprotec process to preprocess used oil for lube oil recovery.

Strathcona refinery is currently using the product from the Shurtleff Waste Oil Refining system as a feed to the cat cracker. Samples from the process showed that the Shurtleff product is consistent over run length and is metals-free but does contain some residual contaminants. Results from this work were used to set levels of Shurtleff product injection into the cat cracker.

Enprotec offers a distillation plus chemical treatment process to upgrade used oil to valuable refinery feedstocks. A team consisting of L&S/ESC and SRC was formed in 4Q93 to assess application of this process for lube recovery in Sarnia. The concept is to process used oil off-site, blend product containing lube-molecules into crude for lube recovery and route fuels products to finished product tankage. Assuming the Enprotec process is successful, a detailed project schedule outlining milestones/timing has been put together to implement this process by mid 1995. Activities are underway towards the first major objective, i.e. assessment of technical feasibility including processibility of used oil products in Sarnia lube plants and judgement on base stock quality.

BASE STOCKS

1. Exxon/Competitive Base Stock Survey

(World Mutual)

Reports generated on request from the base stock database include:

- 1) A review of hydrocracked base stocks, used by ECI to help establish a new API category for 120 VI+ base stocks such as EXXSYN;
- 2) An information package on rerefined base stocks, presented by EUSA to the ASTM base stock specifications task force with the intent of convincing industry of the need for controls on rerefined base stocks;
- 3) Correlations between volatility at 375°C and 371°C that were used to define appropriate JY specifications for volatility at 371°C without the need for additional laboratory work.

2. Technical Support for ECI Crude Approval Program

(World Mutual)

Assistance was provided to ECI in assessing Ekofisk and Hudson crudes (North Sea) for lubes production. These crudes have high basic nitrogen contents, causing concern for base stock oxidation stability in industrial oil formulations, in particular ASTM D943 life in the Teresso formulation. With the co-operation of the lube assay team, samples of 150N and 600N base stocks were prepared at various VI levels. D943 oxidation screener tests are being run to help ECI decide on appropriate VI levels for the Ekofisk and Hudson base stocks.

In future, assays will include a D943 oxidation screener in order to streamline approval of high basic nitrogen content crudes of interest to ECI. Two screener candidates are being evaluated, the rotary bomb test run at 120°C with the full Teresso adpack, and a D943 type test run with 1/10th the normal Teresso adpack concentration. This work will finish in early 1994 when a recommendation will be made on which test to incorporate into the assay. Either screener has the potential to shorten approval time by several months.

3. Hydrotreated Base Stock Performance

(World Mutual)

In the early 1980's, low base stock sulphur content was established as being a debit in the Sequence IIIE engine test. This has been a barrier to the application of severe hydroprocessing technology for lubes within the Exxon circuit. However, with the development of additive technology to meet more demanding engine oil performance requirements, there have been grounds for believing that the importance of base stock sulphur has diminished. Advantage was taken of a Baytown refinery test of severe HYDROFINING (VI Hop) technology to test this belief. A 3 ppm sulphur content 100N base stock was produced from the Baytown test product and formulated as a 5W30 using Paramins SH technology. This gave an outstanding result of 82 hours to 375% viscosity increase in the IIIE test, which compares favourably to results on similarly formulated commercial base stocks, including those with sulphur contents as high as 0.5 wt%. This confirms that low sulphur content is no longer an engine oil performance issue for severely hydrotreated base stocks.

Also in this program, samples of base stock were prepared for toxicity studies by extracting a mixture of virgin and hydrocracked distillate. These are being analyzed by DMSO extraction and run in modified Ames tests in order to better understand toxicity issues around the use of hydrotreated fuels streams for lube base stock production.

4. MXT 5 Yield Maximization

(Imperial Oil, Products)

Two projects were initiated in 1993 to maximize MXT 5 production at the Sarnia hydrocracker (HCIS). The first was to assess the impact of replacing the current hydrocracking catalyst with IFP's catalyst HYC-642 (see 'Refining Support' section) and the second was development of a model to correlate MXT 5 yield/quality with HCIS operation. With this capability, HCIS operation can be optimized to maximize MXT 5 yields; a 5% increase in MXT 5 is worth 400 k\$/yr.

Due to intermediate tankage between hydrocracker recycle production and MXT 5 production, commercial data could not be utilized to develop a MXT 5 predictor model. Efforts are now focussed on utilizing the data available for hydrocracker recycle samples worked up in the laboratory. A data base with hydrocracker operational data, recycle quality and MXT 5 yield/quality has been developed. Analyses to-date have shown that several recycle quality parameters, e.g. mid boiling point, impact MXT 5 yield/quality significantly. Correlations are being tested for statistical significance. The next step would be to incorporate hydrocracker operating data to correlate MXT 5 yield/quality with hydrocracker operation.

5. Crude Flexibility

(Imperial Oil, Products)

One of the most important factors in reducing costs in refinery operation is crude selection. However, it is equally important that product quality not be compromised. Work was carried out this year to determine the lube potential of a number of new crudes and to

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(Imperial Oil, Products)

One of the most important factors in reducing costs in refinery operation is crude selection. However, it is equally important that product quality not be compromised. Work was carried out this year to determine the lube potential of a number of new crudes and to

determine the impact of increasing the amount of sour and/or equity crude in the refinery slate. Results showed that Husky crude has little value for lubes while another upgraded crude, Newgrade B, had reasonably good yields of 5 and 10 grade molecules. Federated was approved at the 100% level for base stock manufacture. Lube assays were also completed on Koch, Bow River and Rainbow crudes.

In order to take advantage of market opportunities, a crude approval protocol was developed that would identify which crudes are suitable for lubes, and state a recommended level at which the crudes can be processed. This protocol has been approved in principle by both refineries.

6. Base Stock Flexibility

(Imperial Oil, Products)

Light lube production continues to be stressed at both refineries. Several projects were undertaken to maximize distillate management:

- A plant test was carried out to inject LVIS bottoms (currently used as cat feed) into regular 10 distillate. This has the potential to increase MCT 10 production by 5% and will free up more days for MCT 5 production.
- Another plant test and SRC results demonstrated that a 95 VI, low volatility, 100N base stock can be made within the normal operating procedures of the refinery. The production of this 100N can significantly increase light lube production. Commercialization is planned for 1Q94.
- Strathcona refinery produces a high flash 80N refinery stream (dewaxed HAGO) which was evaluated as a lube feedstock. It was demonstrated that an acceptable electrical oil could be formulated with this material and that it was suitable for products that require excellent low temperature properties.
- Evaluation of the top-pump-around-stream of LVIS showed that it could be used as a process oil. This has now been commercialized and is designated by the base stock name XT-50.

7. Base Stock Quality

(Imperial Oil, Products)

New specifications were issued for MCT 5, MCT 10, and MCT 30 which are compatible with API interchange guidelines and plant capabilities.

A protocol to manage change in base stock manufacture and use was also introduced during the year. This protocol ensures that any changes are supported by Research results that will determine any impact on product quality. As well, the protocol will ensure that all stakeholders are aware and approve of any changes.

(Imperial Oil, Products)

8. Technical Support

During 1993, some of the more significant technical support contributions from the base stock group were as follows.

- An investigation of the cause of the odour in Paraffin 5 was carried out. Although a definitive origin of the odour was not found it was determined that increasing the hydrofiner severity did decrease the odour level. As a result Sarnia refinery has adjusted its operating conditions.
- Monitoring of re-refined oils that are supplied to IOL showed that the PNA content of these oils has dropped over the last year. This data gives increased confidence in the use of these base stocks in ENTECH products.
- A statistical analysis of plant data on base stock properties was undertaken to determine plant variability. This data will be used to ensure product formulations are robust.
- A matrix of experiments demonstrated that MCT 60 will darken on storage regardless of storage conditions. Base stock manufacturing specifications are being adjusted which will improve storage stability.
- A test to measure low levels of phenol in base stock was developed. This will allow IOP to meet external customer requirements.
- Technical presentations to both internal and external customers on factors affecting base stock quality have continued.

CUSTOMER SUPPORT

(Imperial Oil, Products)

"Attention to technical and service differentiation and a high degree of direct customer contact are two key ingredients necessary to develop the strength of the Esso brand". This statement, found in the Lubricants and Specialties, Strategic Business Review is what drives the activities of the Customer Support group.

Performed in harmony with the strategies of the various IOP Marketing groups, activities involve customer training, used oil analysis, competitive product analysis, lubricant application problem solving and failure analysis. In addition, over 18% of the group's time has been devoted to direct contact with our customers and sales force through the provision of technical information and counsel.

Demand from customers for used oil analysis continues to grow with the year end sample volume exceeding 7700. Over 70 % of these samples were from the Esso Security Plus (ESP) program. The sales force have effectively used ESP as a value-added, lubrication service with over 175 customers currently enrolled in the program.

To meet the increasing need for reduced cost and faster response, a complete review of work processes was conducted and changes initiated. Key elements of these changes consisted of commissioning of a new sample management system, introduction of automation through auto samplers and bar-coding as well as a physical redesign of the laboratory to match the newly mapped work process developed by the group.

In the area of customer training, five lubrication seminars were held in 1993 involving 127 customers. Target customer groups included purchasing agents, oil and gas production and natural gas transmission. A seminar was also held in Sudbury for INCO Ltd. The seminars have proven to be an excellent vehicle to educate our customers in all areas of lubrication and product applications. In addition, they are a source of information on customer needs and expectations which feed both the marketing business channels and the product development process.

27 specific lubrication projects were completed for Marketing and their customers. They involved the analysis of a variety of failed parts, fundamental lubrication calculations and product application problems. A technical network of experts within Research and Marketing Technical Services was utilized in the analysis and presentation of these projects.

REFINING AND ENVIRONMENTAL

OVERVIEW

The Refining and Environmental Sections report directly to the SRC Manager. The main focus of the work in these sections continues to be to provide technical support to Imperial Oil, Products Division with expertise in applying refining technology, environmental monitoring, site remediation, water treatment and waste handling. Some work is carried out for Imperial Oil, Resources Division on the processing of heavy crudes. Contract work for other Exxon affiliates is also done from time to time as needs develop.

The general direction of the research projects is set each fall through meetings with the business sponsors. This ensures alignment with strategic thrusts and customer needs. However, in addition, individual projects are planned jointly with customers on an ongoing basis throughout the year. Financial incentives for each project are provided by the customer to ensure the work is clearly justified and prioritized. In the Internal Market Economy (IME), specific contracts which define the project objectives, deliverables and budget are developed with each customer.

The following technology highlights indicate the milestones reached in 1993 and the status of projects continuing into 1994.

REFINING SUPPORT

1. Operations Support

(Imperial Oil, Products - Refineries)

The refining-sponsored operations support program addressed more than 20 specific requests for technical assistance for Imperial Oil, Products Division refineries (Sarnia, Nanticoke, Dartmouth, Strathcona, Ioco) in 1993. Emphasis was on improved customer service in the areas of data quality and turnaround time. The bulk of the requests focussed on spent catalyst (Powerformer, hydrotreater) characterization and detailed analysis of equipment foulant/coking materials.

2. Elemental Sulphur Removal from Mogas

(Imperial Oil, Products - Supply)

Continued development of an Aqueous Caustic Mercaptan (ACM) process for removing elemental sulphur from pipeline gasoline focussed on extensive fluid-dynamic testing and several extended pilot unit runs as well as lab kinetic studies. It was found in an extended pilot run, performed to simulate the process, that the target product spec of 3 mg/L or less of elemental sulphur was achieved up to only 250 capacity factor (barrels of gasoline processed per barrel of recirculating caustic), about one-half the expected target of 500 capacity factor. As well, measurement of caustic inventory in the dynamic mixer at shut down showed unusually low levels, indicating inadequate mixing. The dynamic mixer was then modified to include side sampling ports in each of the four stages for measurement of caustic hold-up and elemental sulphur levels. Also, a glass mixer was used for visual observation of the mixing efficiency, including tests of varying the internals, inlet/outlet configurations and start-up procedures.

A critical finding from these fluid-dynamic tests was that dispersing gasoline in caustic resulted in far superior elemental sulphur removal, more uniform mixing and less caustic entrainment than with caustic dispersed (original design). The greater surface area of the smaller, more stable gasoline droplets allows for the increased reaction rates.

Follow-up extended runs were performed which showed that 500 capacity factor could be reached with elemental sulphur levels meeting the target spec. Above this value, phase inversion took place (caustic became dispersed in gasoline). Phase inversion was found to be a function of capacity factor and treat ratio, with the important physical parameter being the caustic to gasoline viscosity ratio. This ratio increases with capacity factor, due to chemical changes in the caustic solution, driving the system towards phase inversion. This can be offset by increasing the caustic treat rate: using a 50% treat rate and up to 750 capacity factor, the elemental sulphur target spec could be met.

Two other methods for removing elemental sulphur from gasoline, namely MTM (Methanolic/Tetramethylammonium hydroxide/Mercaptan) and MCM (Methanolic/Caustic/Mercaptan) were evaluated as possible lower capital cost options to the ACM Treater. Both methods involve injection of small amounts of reagents with high water content which produces two liquid phases. The methanol acts as a phase transfer agent that accelerates sulphur removal from gasoline. Elemental sulphur reduction to the target level

was achieved in both cases. Process development work is focussed on the MCM method since the MTM reagent costs were found to be unacceptably high. Also, the use of an alternative contacting (no mechanical mixing) method, namely Merichem's fibre-film technology, is being evaluated for use with the ACM reagents. Since no dispersion of one phase in the other takes place, Merichem technology has the potential to reduce the number of downstream vessels currently part of the ACM process design.

3. Resid Ultrafiltration (RUF)

(Imperial Oil, Products/ERDL)

3.1 Strathcona RUF Project

Resid Ultrafiltration (RUF) plant tests at Strathcona LVPS have shown a decline in the permeation rate over time due to membrane fouling. Preliminary findings indicate that trace amounts of iron sulphide foul the membrane pores. Plant tests on 1" diameter elements from CeraMem have shown that a packed bed of gamma-alumina and 225 μm Pall filters reduce the rate of fouling, with the packed bed being the lowest cost option. The amount of foulant per square foot of membrane (dose) also had a significant effect. Foulant doses are expected to be significantly lower for the 6" diameter commercial elements compared to the 1" elements.

Commercial elements (50 \AA gamma-alumina) from CeraMem were tested at Strathcona in resid service with a gamma-alumina packed-bed pretreater. In the initial two runs, elements cracked during start-up due to thermal expansion differences between the packing gland housing and the element. A face-seal housing with internal bellows to accommodate the thermal expansion eliminated the start-up failures. However, two elements failed during operation due to high transmembrane pressure. Procedural and process control changes eliminated the transmembrane pressure failures. In the last run (which was terminated after 16 days due to a planned pipestill turnaround) a low flux decline of 0-0.7% per day was observed.

Another test started in December, 1993 with the 6" diameter commercial element to demonstrate technical readiness, and will continue for approximately 2 months.

3.2 New Ultrafiltration Applications

(Imperial Oil, Products/ER&E)

Large incentives were identified by Sakai to recover vacuum gas oil from spent wash oil. Ultrafiltration pilot tests on Sakai spent wash oil showed a 51% Ni+V rejection (amount of metals remaining in the feed) at the expected commercial operating conditions. More work is planned in 1994 at ERDL (Baton Rouge lab) to support the Sakai wash oil application.

IOL refineries are processing cheaper crudes as part of a cost-cutting challenge. Since these crudes produce more pitch than conventional crudes, there is an opportunity to recover additional cat feed from pitch using CeraMem filters. The pilot tests with Dartmouth, Nanticoke and Strathcona pitch showed that permeation rate increased with decreasing viscosity while the rejection performances were similar. The Ni+V, MCR, nitrogen and sulphur rejections were 50, 28, 19 and 8 percent, respectively, with the viscosity

reduction increasing with increasing feed viscosity. An economic study for a Strathcona SRC in 1994. application showed less than 10% DCF. No further work on resid ultrafiltration is planned at

4. HCN Membrane Development

(World Mutual - ERDL)

Polyester imide (PEI) membrane prepolymer synthesis time was increased from 4 hours to 6 hours for improvement of long term membrane stability. Elements manufactured with the 6 hour prepolymer-based membrane were tested in FOS refinery during 1Q93 and showed excellent flux and selectivity maintenance. With the success of the FOS plant test, the HCN membrane pervaporation program focussed on evaluating and improving quality control in both membrane and element fabrication. In association with Desalination Systems Inc. (DSI), SRC was responsible for the membrane portion of this program.

Membrane Quality Control (QC) spots can be classified as either imperfections or defects, where the former do not show detrimental performance while the latter do. Pre-conditioning of the membrane allows imperfections to be readily distinguished from defects. However, scanning electron micrograph analysis of imperfections and defects showed that they have similar features. Therefore, it was decided that all QC spots should be patched with epoxy during the membrane QC procedure. This conservative approach is feasible in commercial production with no increase in production costs or significant loss of effective membrane area. Membrane in the FOS plant test elements was treated in this manner. Inspection of the used FOS elements showed the membrane to be in excellent condition.

A formal quality control program for polymer production, membrane production and element fabrication was conducted. DSI, Exxon Research and Development Laboratory (ERDL), SRC and Exxon Engineering jointly established the production and testing methods to be used. Then, with Exxon only present to audit the work, DSI produced six independent batches of polymer, four independent membrane casts and 20 elements according to these specifications. Extensive lab membrane testing at DSI and SRC and element testing in the ERDL pilot facility have established the degree of reproducibility at each stage of the fabrication process. Polymer and membrane production proved very reproducible. Element testing is still in progress and results to date look very good.

5. HCIS R-2 Catalyst Evaluation

(Imperial Oil, Products - Supply)

IFP HYC-642 hydrocracking catalyst is currently undergoing extensive evaluation as follow-up to a 1992 scoping study in a pilot hydrocracker using heavy coker gas oil as feedstock. The purpose of this second evaluation is to get sufficient data to support a possible commercialization of the IFP catalyst at the Sarnia HCIS unit in November 1994. Pilot operating conditions evaluated to date cover typical commercial summer/winter operations and the optimum conditions for maximum lubes product volumes. The pilot run also includes an evaluation of the UOP HC-8 catalyst, currently used in the Sarnia commercial unit, at appropriate conditions to establish a pilot base case for comparison purposes. Results obtained to date for the IFP catalyst indicate substantial lube (MXT 5) yield benefit (previous work focussed on LXT yield). Fuels distillate yield was also increased

at the expense of gasoline production, while both fuels and lubes distillate qualities were also improved. A large sample of MXT 5 basestock was produced using the IFP catalyst for evaluation in engine and industrial oils to assure that the required performance specifications are met. Those evaluations showed that all the base oil and formulation requirements are met within the existing protocol. The results from the pilot evaluations will be used to set the project profitability economics and facility modification requirements.

Statistical analysis of pilot plant data was initiated to develop both pilot and commercial guidelines for use of IFP catalyst. Initial statistical work is focussed on lube production from the recycle stream where R-2 reactor and cut-point temperatures were identified as critical factors affecting the quality and yields of MXT 5 distillate. The predicted values (calculated using the statistical model) of VI, volatility and yield for MXT 5 showed excellent agreement with the experimental pilot plant data. Within the limited envelope of operating conditions tested, any additional input related to the performance of IFP catalyst for lubes production can be predicted from the statistical model without need to run additional pilot plant tests. Future work will expand the statistical correlations to include fuels and other operating parameters.

6. Oligomerization of Propylene over ZSM-5 (Exxon Chemicals/IO Chemicals)

Exxon Chemicals, Intermediates Technology, is assessing the feasibility and attractiveness of replacing existing solid phosphoric acid (SPA) catalyst used in higher olefins units with a zeolite catalyst. The expected benefits for zeolite over SPA arise from higher value product mix, namely better selectivity to heavy Isopar feed oligomers, and reduced catalyst disposal costs. Previous ZSM-5 catalyst testing in an IT-Machelen tubular pilot unit showed attractive C13+ selectivities (30% vs 5-7% on SPA) with high propylene conversion.

Further pilot testing was initiated at SRC where initial testing involved replicating the Machelen test results and screening the effect of operating pressure. The results showed similar product selectivity and conversion levels as those obtained in previous runs at Machelen. Propylene conversions were maintained in the 90-95% range for up to 1,350 cat hours and up to 320°C. Correspondingly, 30-35% selectivity to higher olefins (418°F+ material) was achieved for up to 1,200 cat hours with temperatures as high as 300°C. Rapid catalyst deactivation occurred above 325°C with conversion levels dropping to 80% and selectivities below 15% at the end of the run (1,419 cat hours). No further work is planned at SRC in support of this Chemicals' project.

7. Upgrading Cat Frac Bottoms (CFB) to Asphalt (Imperial Oil, Products-Supply)

CFB is too soft to meet road asphalt specifications. Blend tests with 85/100 Cold Lake pen grade asphalt indicate that a 150/200 penetration product can be made using 15 to 22 wt% CFB but the viscosity of the blends is below A grade prairie specifications. The effect of mixing, air oxidation, sulfonation and nitration on Cold Lake/CFB blends was investigated. The data can be used to produce an A grade 85/100 pen grade product.

The incentives to upgrade CFB at Strathcona have dropped since new outlets for CFB have been found. As a result, no further work is planned in this area in 1994.

8. Heavy Crude Upgrading

8.1 Syn crude Synthetic Crude Oil Characterization Program

(Syncrude)

A detailed characterization was carried out on Syncrude Synthetic Crude Oil (SCO) samples to assess potential contamination during pipelining. Similar samples of "as produced" SCO from Syncrude and "as received" SCO sampled at Interprovincial Pipe Line in Marysville, Michigan were sent to SRC for characterization. Samples of the three rundown streams which make up SCO (naphtha, light gas oil and heavy gas oil) were also analyzed in order to fully characterize these streams and optimize the SCO blend's market potential. The results indicated that the most significant difference between the before- and after-pipelined crudes for this particular shipment was the 1.2 wt% additional bottoms in the pipelined material.

8.2 Residfining of Hydrovisbroken Athabasca Feedstock (Imperial Oil, Resources)

A pilot Residfining program over RT-621 catalyst was carried out using an Emulsion Hydrovisbroken (EHVB) Athabasca feedstock. This feedstock was the product of an upgrading scheme intended to achieve a premium resid product fraction. The purpose of the hydrotreating run was to assess the effects of space velocity (0.27 and 0.1 LHSV) on product quality at a 390°C run temperature. Also, the 0.27 LHSV product was fractionated and characterized.

The results indicated that reducing space velocity from 0.27 to 0.1 significantly enhanced HDN activity and moderately increased MCR removal. HDS was extensive at 0.27 LHSV and reducing the space velocity to 0.1 achieved almost complete desulphurization. There was no gain in nickel and vanadium metal removal with the reduction in space velocity, since both were virtually completely removed at the higher level. The conversion levels of both the 343°C+ and 565°C+ fractions were fairly significant at 0.27 LHSV and increased further when the space velocity was reduced. A second Residfining run is underway with an Emulsion Visbroken (EVB) Athabasca feedstock from the same upgrading scheme, with the objective of running at lower temperatures in order to minimize the 565°C+ conversion while maintaining resid sulphur content less than 0.5 wt%.

8.3 Characterization of Bitumen Product from a Sodium Metal Upgrading Scheme

(Imperial Oil, Resources)

A detailed characterization study was undertaken on Cold Lake and Athabasca Bitumen feedstocks and products from a sodium metal desulphurization process. The process involves the addition of sodium metal to the bitumen at high temperatures (343°C+) in the presence of hydrogen (~200 psig), resulting in deep desulphurization of the crude. Total product sulphur levels of less than 0.2 wt% are achievable. Detailed feedstock and product characterizations are being done in order to quantify the effect of process variables on product quality. Although Exxon Research Development Laboratory demonstrated the feasibility of the sodium metal desulphurization process several years ago, no detailed product

characterization work was performed at that time. This current study will help to understand the benefits of this upgrading method in light of advances in electrolytic sodium recovery methods and improved process economics.

(Imperial Oil, Resources)

9. Porous-Walled (Membrane) Reactor

The membrane reactor was proposed as a high efficiency contacting device that will minimize diffusional resistances, thereby offering the potential of higher catalyst activity and higher product selectivities (lower gas make) in heavy crude upgrading as well as hydroprocessing of fuels and lubes.

Residfining of a Cold Lake vacuum gas oil was used for demonstration of the concept. Two membrane reactor modes were tested. The first was a "sparged" mode where a porous, ceramic membrane was installed concentrically inside a tubular reactor and filled with conventional catalyst pellets. The second was a "thin film" mode where an identical porous, ceramic membrane was installed and a thin layer (5-10 micron) of catalyst was deposited on the inner membrane surface. In both cases, hydrogen is introduced from the shell side of the reactor through the membrane surface, effectively resulting in an infinite number of hydrogen injection points along the length of the reactor. Both reactor modes were compared with conventional packed bed operation at the same operating conditions and geometry.

Operability of the "sparged" reactor mode has been demonstrated with moderate improvement in HDS relative to "conventional" packed bed reactor operation. "Thin film" reactor mode testing, where the most significant benefits are expected, is in progress. Thin film catalyst coating techniques were developed at Exxon Research Development Laboratory (ERDL) for this project, resulting in a catalyst layer that is 5 -10 microns in thickness which represents a diffusion path length only 1% that of a conventional hydroprocessing catalyst particle.

10. Benicia Clean Fuels Project

(Benicia Refinery)

A pilot hydrocracking study was undertaken to provide data to support ER&E model predictions used to establish a design basis for modifications to the Benicia hydro-cracker. In order to confirm that the pilot unit could adequately represent the commercial unit, a base case evaluation was carried out, simulating the current operating conditions of the Benicia hydrocracker [HDN-60 (NiMo) HDN catalyst in R-1 and HC-28 (Pd on molecular sieve) in R-2]. The hydrocracking pilot unit was operated in MOGAS mode with extinction simulating the commercial unit, based on the limited data available for comparison.

The design basis was replacement of all diesel and light atmospheric gas oil feed to the hydrocracker with heavy-heavy cat naphtha and heavy-heavy powerformate, two streams that would be created with refinery modifications. Also, the product fractionator cut-point was decreased to 155°C. For this evaluation, a catalyst that had been aged in the commercial unit was used for R-1 and R-2 to simulate EOR (end-of-run) activities and to

generate EOR product yields and qualities and hydrogen consumption. The pilot study confirmed most of the model predictions and a hybrid of the two data sources was created (by ER&E) for the design basis.

11. Sulphur Speciation Study for the Sarnia Coker

(Sarnia Refinery)

Omnicon Consultants and SRC undertook a joint program to develop an analytical tool for sulphur speciation aimed at determining crude quality and facilitating prediction of corrosion rates. Samples were collected around the Combo-Coker atmospheric pipestill (CCIS APS) and analyzed by conventional wet chemical methods at Baytown and by new mass spectrometry-based methods at Omnicon (PMD-SIM) and SRC (GC-SIM). The Baytown results were used to calibrate the MS methods. Both methods as well as a Total Reactive Sulphur (TRS) MS method developed at SRC appear promising. Details of the SRC analytical method development are presented in the "Analytical" section of this report.

12. Cat Cracker Advanced Process Modelling

(Imperial Oil, Products - Supply)

Detailed characterization of crudes and cat cracker streams was carried out to get better compositional data as input to the Advanced Process Model, in order to more accurately evaluate potential cat cracker feedstocks and process/hardware modifications. This work focused on the evaluation of cat cracker "short contact time" modifications, the quality of resid ultrafiltration permeate as cat cracker feedstock, and crude screening.

ENVIRONMENTAL TECHNOLOGY AND SERVICES

1. Waste Treatment

(Imperial Oil Products and Chemicals)

Landfarming is the most cost-effective technology currently available to IOL for the treatment of oily and biological sludges (~\$30/tonne versus >\$100/tonne for other treatment methods). An action team, with SRC participation, has completed a performance assessment to ensure effective utilization of the existing landfarm at Dartmouth Refinery. Several areas of concern were identified: high levels of hydrocarbon, non-optimum soil pH and poor drainage characteristics. Lab studies showed that the soil contains 1.5-3.0 wt % biorefractory hydrocarbons which directionally lower the biodegradation rate relative to background soil which contains no biorefractory material. Experiments showed that use of straw as a topsoil amendment increased the soil hydraulic conductivity, which should improve drainage and help avoid anaerobic soil conditions which hinder biodegradation. Dartmouth is continuing to implement operational changes to improve landfarm performance.

A joint study with Corporate Research has been undertaken to assess the potential of advanced nutrient packages to enhance the rate of biodegradation in landfarms, thereby increasing capacity and reliability of operation. A series of slow release and oleophilic fertilizers, dispersants and surfactant clathrates are being evaluated with soil and wastes from the Sarnia Refinery landfarm. The experimental program is complete and data analysis will be concluded by year-end.

Higher Olefins units at Sarnia and Dartmouth required new disposal options for spent phosphoric acid polycatalyst. Spent polycatalyst is currently stored in Dartmouth's secure landfill, while Sarnia's spent catalyst was (until recently) sent to Laidlaw's secure landfill for disposal at a cost of \$440/tonne. Research has been actively involved over the past two years in the development of alternate disposal options for the waste materials. Work with Philip Environmental identified a recycle option in which acidic spent polycatalyst (high phosphorus content) is processed with alkaline cement kiln dust (high potassium content) to produce a neutralized PK fertilizer product suitable for agricultural use. Using technical data provided by SRC, Philip obtained Ontario Ministry of Agriculture approval to market their new product. After a pilot test, Sarnia began shipping spent polycatalyst to Philip as a fertilizer feedstock on a routine basis in 4Q93. Processing costs (\$80/tonne) are roughly one-sixth those of secure landfill. This reuse option is economically and environmentally superior to disposal in secure landfill. Dartmouth is currently negotiating with Philip to accept both their ongoing polycatalyst production and the material (approximately 800 tonnes) stored in the secure landfill. This reuse option has potentially wide applicability within Exxon.

The Sarnia Chemicals Plant spent 2.6 M\$ for the disposal of hazardous waste in the 1990-92 time frame. There was a general trend in reduction of solid and liquid wastes produced by the plant; however, disposal costs increased from 1990 to 1992, mainly due to increased unit disposal costs at Laidlaw. Analysis of data from the waste information system showed that disposal costs accounted for 60% of total waste handling costs, while transportation and bucket rentals accounted for the remaining 40%. The Chemical Plant is currently improving its bucket management system in order to capture significant reductions in rental fees. The largest waste streams produced (by volume, cost, and business unit) were identified. The Chemicals Plant, with SRC assistance, is actively exploring reduction/recycling opportunities for these waste streams that should result in the reduction of current disposal costs and potential future liabilities. Emphasis is being focused on spent sulphidic caustic and waste MEA/water streams.

Disposal of refinery wastes at secure landfill sites is expensive and entails the risk of potential future liabilities, making waste reduction/recycling a desirable corporate goal. In 1992, SRC performed a study with 1991 data that identified high disposal cost waste streams at the Sarnia Refinery. With SRC assistance (especially ALIS and soil disposal), the Refinery reduced the cost of hazardous waste disposal for 1992 by 54% (1584 to 730 k\$). An analysis was performed on 1992 data to identify further cost reduction opportunities. Bucket rental costs were reduced only marginally, and the Refinery is currently improving its bucket management system in order to capture significant cost savings. SRC is working with the Refinery to find alternate reduction/recycling opportunities for high cost/volume waste streams, such as spent caustic, identified by the study.

Audits of three multi-party waste management facilities have been completed for the Marketing Department. Taro Aggregates (Philip Environmental), Niagara Waste Systems (Walker Industries), and Ridge Landfill (Browning Ferris Industries) all scored as "Good" using the ER&E guidelines meaning that they are acceptable for use by Imperial Oil.

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These audits are required by Operations Integrity to ensure that Imperial Oil's wastes are being handled in a manner that is consistent and compatible with their policies and business objectives.

2. Clean-up of Contaminated Sites

(IOP, ER&E, MERD)

The former Texaco Refinery site in Montreal contains 400 k tonnes of hydrocarbon-contaminated soil. The site was slated for incineration, in a joint project with Petro-Canada, at a cost of \$100/tonne. Petro-Canada's withdrawal from the project, together with a relaxation in clean-up criteria, has provided an opportunity to bioremediate the site with the potential for significant cost savings (~\$50/tonne). Soils from the site are being subjected to a joint ER&E/Corporate Research/SRC biotreatability study funded by IOL and the World Mutual R&D program. The study has been integrated with a similar on-going program for Bayway Refinery. The latest leads in oleophilic (e.g. INIPOL) and slow release fertilizers are being tested to determine the best candidates for field trials. Results to date indicate that: (1) bioremediation can achieve 50-60% hydrocarbon degradation required to meet regulatory requirements, (2) the oleophilic fertilizer is generally the best performer, and (3) the degradation rate is independent of soil and hydrocarbon type as well as the starting level of hydrocarbons in the soil. Results from field trials (using 500 m³ biopiles) which started mid-summer confirm the lab results.

A side-by-side comparison study was carried out with selected bioremediation technology vendors (Makhteshim, Grace Dearborn, Mycotech and NAT Environmental) to ensure that the best technological advances are used. After 20 weeks of testing, an in-house oleophilic fertilizer developed by Corporate Research is equivalent to the best of competition.

Remediation/disposal of contaminated soil from active retail sites is constrained by the need to minimize disruption to customers and to reduce/eliminate long term liability for contaminated soils at landfills or treatment sites not controlled by IOL/Exxon. An engineered, in-ground vapour extraction/bioremediation system has been developed in conjunction with ER&E as part of the Marketing Engineering R&D (MERD) program. Contaminated soil is removed and conditioned to promote biological activity of the indigenous microorganism and placed in an engineered "bio-pocket" below grade. The vapour extraction system is operated until volatile emissions reach an asymptote at which time bioremediation is initiated by addition of nutrients and turndown of the vapour extraction blower to aerate the soil. An estimate for a site in London, Ontario showed costs to be in the 40-50 k\$/m³ range which is equivalent to landfill tipping fees. The bio-pocket was installed at the London site in conjunction with the Marketing Department of IOP during August/September. The on-site work was directed by use of a portable gas chromatograph which provided real-time information regarding soil contamination levels. Vapour extraction was started in late September with off-gas treatment using a thermal/catalytic oxidation unit. After one month of operation, the hydrocarbon vapour levels were reduced to a low level and treatment was no longer required. The bioremediation phase is on-going and will be monitored closely in 1994.

Large volumes of contaminated soil (50-1500 tonnes) are encountered at hundreds of sites each year during tank replacement at active retail sites or remediation activities at inactive sites. Current methods of hydrocarbon measurement involving inaccurate

The former Texaco Refinery site in Montreal contains 400 k tonnes of hydrocarbon-contaminated soil. The site was slated for incineration, in a joint project with Petro-Canada, at a cost of \$100/tonne. Petro-Canada's withdrawal from the project, together with a relaxation in clean-up criteria, has provided an opportunity to bioremediate the site with the potential for significant cost savings (~\$50/tonne). Soils from the site are being subjected to a joint ER&E/Corporate Research/SRC biotreatability study funded by IOL and the World Mutual R&D program. The study has been integrated with a similar on-going program for Bayway Refinery. The latest leads in oleophilic (e.g. INIPOL) and slow release fertilizers are being tested to determine the best candidates for field trials. Results to date indicate that: (1) bioremediation can achieve 50-60% hydrocarbon degradation required to meet regulatory requirements, (2) the oleophilic fertilizer is generally the best performer, and (3) the degradation rate is independent of soil and hydrocarbon type as well as the starting level of hydrocarbons in the soil. Results from field trials (using 500 m³ biopiles) which started mid-summer confirm the lab results.

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field screening methods (hydrocarbon vapour, odour) can lead to removal/treatment of excessive volumes of soil at costs ranging from \$50-1500/tonne, adding significantly to site restoration costs. To meet the need for "real-time" analysis, methods for simultaneous analysis of BTEX and TPH have been developed using a field-portable GC (MERD funding). A "solvent-less" thermal desorption method has been developed which quickly/quantitatively removes mogas/diesel from soil matrices. A secondary trap system has been developed, in co-operation with the GC manufacturer, which eliminates the interference of soil water with BTEX quantification. The TD method has improved ease of use in the field and eliminates the use of chlorinated solvents. A cross-check with conventional laboratory analysis methods is on-going.

A detailed site assessment has been completed and a remediation plan developed for the leased and owned sections of the Ashbridges Bay site on the Toronto Harbourfront in support of negotiations with the City of Toronto regarding a possible takeover of both properties by the city.

Dense non-aqueous phase liquids (DNAPL) have been found in groundwater monitoring wells at the Blending & Grease plant on the Toronto Harbourfront. Extensive characterization of the hydrocarbons in the wells identified coal tar from the carburetted water gas process. The DNAPL is very similar in composition to material found on other properties in the area and is most likely the result of the landfill operation used to create the sites in the early 1900's.

3. Wastewater Treatment

(Imperial Oil Products and Chemicals)

As part of Operations Integrity initiatives, refinery Wastewater Effluent Treatment Systems (WETS) must be statistically capable of meeting local water effluent quality regulations/guidelines. A Refinery/SRC capability analysis of Dartmouth WETS identified areas of incapability which require follow-up studies to identify/implement steps to improve capability levels. SRC is assisting the Refinery lab to assess existing environmental analytical methods (minimum detection limits, test methods and standard operating procedures) to ensure their statistical capability. A study of the solids/oil removal efficiencies of the pressure dual media filters led to operational changes which resulted in a step change improvement in solids removal efficiency. A similar study has shown low removal efficiencies across the separators. Follow-up studies to determine the cause are planned for 1Q94. Lab testing showed that the solids/oil removal efficiency of the dissolved air flotation unit could be greatly improved by chemical addition. A confirmatory plant test is planned in 1Q94. A preliminary source control program identified surface water runoff as the major source of solids/oil to the WETS. Identification of specific sources and implementation of reduction strategies will reduce the load on the WETS. SRC will continue to work closely with the Refinery in 1H94 to develop/implement systems necessary to achieve a statistically capable WETS.

A review of the performance of Strathcona's wastewater treatment plant (WWTP) was conducted to assess possible compliance issues with respect to the new water licence issued in late 1992. A statistical analysis of 1992 data showed that the WWTP was

fully capable of meeting all discharge limits for pH and phenol, marginally to fully capable for suspended solids, chemical oxygen demand, ammonia and oil and grease, and incapable for sulphides. An assessment of the performance of the various units of the WWTP identified possible areas for operational/facility enhancements which should improve performance for incapable parameters. Finally, assistance was provided to Strathcona in developing a source WWTP units. SRC is providing ongoing assistance to aid Strathcona in reaching their goal to be statistically capable of meeting all effluent quality criteria.

Tightening regulations in Ontario and British Columbia may force water effluent from commercial agencies to municipal sewers to meet stricter discharge limits in the near future. Characterization of wastewater effluents from oil/water separators at twenty-three agencies in 1991 showed that the effluent exceeded generally acceptable levels for oil/grease, suspended solids, phenolics and benzene/toluene/xylene. A 1992 field test of a commercial filtration/carbon adsorption system at an agency site showed this to be the best treatment technology. In order to obtain information on the treatment costs, the unit was brought to Sarnia Refinery for testing. In two months of operation, the unit produced effluent that was statistically capable of meeting criteria for all parameters, except suspended solids. It is believed the solids incapability is related to the manner in which the unit was operated and not any inherent short-coming in the technology. The test was terminated due to plugging of the carbon absorber; organics breakthrough was not observed. The cost for purchase/disposal of filter and activated carbon elements used by the unit were estimated to be 1.6-2.3¢/litre of water treated. Greater than 50% of the cost was related to purchase/disposal of filter elements. This cost will be site specific and quite variable. In cases where toxicity or dissolved organics are of concern, this may be the preferred technology for secondary effluent treatment.

Ontario legislation dictates that in 1994 the wastewater effluents from the petroleum sector will have to pass a toxicity test using daphnia magna. A study is ongoing using portable flowmeters and autosamplers to assess the water effluent from the various process units within the Sarnia Chemical Plant. Work to date has pin-pointed two streams which contribute significantly to effluent loading. A program is being formulated to reduce the toxicological impact of these streams.

Clean water effluent from the Sarnia Chemical Plant is treated for suspended solids by means of an impounding basin before discharge. Operational data indicated that the suspended solids concentration in the effluent is increasing rapidly. A profile of the basin completed by SRC concluded that solids accumulation has reduced the residence time to 4-5 minutes. A scope of work/bid contract has been prepared by SRC/Chem Plant personnel for an excavation operation to begin 1Q94.

Environmental compliance with British Columbia provincial regulations regarding effluent toxicity towards trout became a problem at Ioco's refinery. On-site investigations revealed that a very sour, light and unsaturated process leak was consuming cooling water chlorination. Chlorine consumption led to accelerated bacterial growth and corrosion in the cooling system and was addressed by a multitude of chemical treatments.

These chemicals, especially the in-situ produced organic chloramines, were likely responsible for the effluent trout toxicity. Isolation of the cooling water from WETS eliminated the problem. Recommendations for future operation were provided.

4. Air Emissions

(Imperial Oil Products and Chemicals)

The National Pollutant Release Inventory (NPRI), modelled after the U.S. Toxics Release Inventory (TRI), became federal regulation in early 1993. Significant users of 176 chemicals will be required to report 1993 releases of these substances to air, water and land in June, 1994. The NPRI implementation strategy involves a broad approach where definitions, standards, methodologies, etc. are maintained consistent across Imperial Oil. A project team was formed, with participants from Chemicals, Refining and Resources Divisions, to develop the systems needed for NPRI. The NPRI effort has been leveraged extensively with ER&E and EUSA reporting sites that have been performing TRI since 1987. SRC has evaluated and recommended emission estimation methods appropriate to IOL, defined the data requirements for the release calculations and created an emission source checklist specifically for refinery sites. A pilot of the NPRI implementation strategy for refineries was completed at Sarnia by a joint Refinery/Refining Technical Services/SRC team. Its purpose was to test and optimize the approach, protocol and tools for collecting the necessary information with minimum impact on refinery personnel. Release and chemical speciation information was collected for process units, tanks, cooling towers, landfarm, water effluent treating system, loading facilities, etc. over a two-week period. Final emission calculations are being performed by Refining Technical Services. This strategy will be used as a model for implementation of NPRI at other refinery sites.

Chemically speciated emissions were calculated from storage tanks for the Sarnia Chemical Plant. A series of spreadsheets was developed to calculate emissions in future. Recommendations were made of inexpensive methods for emissions reduction.

A team has been set up to investigate the causes of opacity at the COB stack in Sarnia Refinery and to identify control options that minimize capital expenditures. A testing program (using a design of experiments approach) to determine the impact of key unit operating variables was completed in December 1992. Analysis of the data showed that the CO boiler was the major contributor to opacity (particulates) and that changes in unit operating variable alone would not result in compliance with the 20% opacity limit on a consistent basis. Subsequent investigations by Refinery/ESC have identified modifications to the CO boiler to improve the efficiency of the coke burning/particulate removal which will occur during the 2Q94 shutdown. SRC will measure emissions pre- and post-shutdown to quantify improvements obtained.

Testing was performed to establish catalyst losses from the regenerator of the Dartmouth FCC which is in the third year of an extended run. The losses (0.9 tonnes/d) were lower than those measured at roughly the same process conditions in 1988. The size distribution data was also very similar, indicating that the cyclone performance at the current high air rate and catalyst inventory is better than in 1988.

The Ioco FCCU is currently in the fourth year of an extended run, with a shutdown scheduled for the fall of 1993. The efficiency of the cyclones appears to be decreasing over time, and particulate concentration leaving the regenerator has been increasing. The particle size data from recent sampling suggests that there is no significant mechanical damage (plugging, holes) but the possibility of refractory roughness appears to fit the data. As the cyclone refractory begins to fragment and tear away from the walls, its efficiency deteriorates and particle losses increase. This accounts for the elevated catalyst losses, but virtually unchanged particle size distributions.

Air dispersion modelling was carried out for Strathcona Refinery to model NO_X, CO and SO₂ emissions under a variety of operation scenarios. The modelling was done in support of ongoing negotiations with Alberta Environment regarding changes in the air permit for the Refinery. Dispersion modelling was also performed for Dartmouth Refinery to determine the impact of crude selection and unit operating conditions on ground level SO₂ emissions.

LUBE PROCESS TECHNOLOGY

OVERVIEW

The Lube Process Technology Division at the Sarnia Research Centre (SRC), in collaboration with its research partners at Exxon Engineering and elsewhere in Exxon, are responsible for the development and application of lube process technology for world-wide Exxon affiliates. This work is funded through the World Mutual system and includes exploratory, development and technology application programs. In addition, billed services are provided to both Exxon customers and prospective licensees of Exxon technology.

The 1993 Lube Process R&D program supported a full range of technologies required for lube basestock manufacture. The program was closely aligned with affiliate business drives through the world mutual R&D planning process. Business alignment was further enhanced by designation of champions for major program deliverables.

The drive to reduce manufacturing costs and increase capacity without significant investment was addressed by a strong STAP effort directed at applying drop-in hardware and process improvements and optimizing plant operations. Other programs emphasized improving correlations and predictive capability, development of advanced analyzers, models and control systems for tighter process control and optimization and completion of testing to determine the technical and commercial viability of membrane dewaxing aid recovery technology. Options for higher product quality were advanced through support for EXXSYN production at the Fawley wax isomerization unit, development of severe HYDROFINING technology and hydroprocessing catalyst screening initiatives. Technology to increase raw material flexibility was advanced through lube assay and basestock characterization activities as well as studies to enhance the production of lubes from fuels hydrocrackers. Support for affiliate used oil initiatives was provided through monitoring of existing and emerging technologies for used oil pretreatment and refinery processing. Efforts to maintain awareness of competitive threats and leveraging opportunities focused on evaluation of Chevron ISODEWAXING technology.

Lube process exploratory research continued to focus on generation of technology that responds to both current and anticipated business needs. Emphasis was placed on strengthening the broad technology base supported by the R&D and STAP programs through increased understanding, on a molecular level, of lube feedstocks and products, as well as the relationship of molecular types to product performance. Specific 1993 initiatives focused on novel extraction solvents, fundamental studies of wax crystallization and opportunities for breakthrough hydroprocessing catalysts. The Science in Lubes Quality (SILQ) initiative continued to focus on development and application of microanalytical methods which have provided valuable new insights into base oil/wax molecular composition and relationship to critical basestock and formulated lube oil performance characteristics.

LUBE ASSAYS AND CORRELATIONS

1. Lube Assays

(World Mutual)

Regional interest to assess new crudes and update lube information on approved crudes continued to be high in 1993. 23 assays were completed (11 new and 12 updates): 12 for ECI, 5 for EUSA and 6 for IOP. Assays for ECI focused on North Sea crudes (new fields and changing blends), Far East crudes (new) and some updates on Middle East and Russian crudes. Assays for EUSA comprised updates on Middle East and mid-continent crudes. Assays carried out for IOP were updates on some Western Canadian crudes and on some new upgraded crude streams.

A new "piggyback" system with Exxon Fuels assays was put into place to ensure that the samples available for a lube assay would be of sufficient quantity to maintain lubes distillation and extraction standards for assay data.

The lube assay program helped to initiate the process of having more crudes either approved or partially approved for lubes. For ECI these included Rabi Kounga, Russian resids from the Ural region and ABU Safah. EUSA also had Rabi Kounga added to their approval list. IOP is considering partial approval for Rainbow and Koch Sour as soon as basestock evaluations are complete. Two North Sea crudes, Ekofisk and Hudson, are undergoing a special evaluation by ECI, due to the relatively high basic nitrogen content of their baseoils.

2. Lubes Correlations

(World Mutual)

Lubes correlation capabilities focused on (a) delivering a tool to help predict relative lube potential from Fuels assay data and (b) assessing the ability of correlations based on molecular data to accurately predict lube processing response. Utilizing Exxon Fuels standard assay data and the LASSY general extraction model, correlations were developed to predict dewaxed oil VI, raffinate dry wax content, and saturates, sulphur and theoretical processing response for a typical 150N distillate. Although the estimates of error are larger than those of the measurement system, the correlations are useful to predict the relative lube potential of new crudes and to predict the effect of a new crude in an already approved crude pool.

Initial studies with a limited number of crudes have demonstrated the potential of using the molecular data from an SRC-developed GC/MS analytical method on whole crudes to predict some lube physical properties. This program will continue on an exploratory basis in 1994.

3. Lubes Predictive Tool

(World Mutual)

The Lubes predictive tool, PC LASSY, was updated with assay data for 25 crudes (new and updates).

Studies to improve PC LASSY predictive capability resulted in the following changes: (a) use of countercurrent extraction data rather than batch extraction data; (b) prediction of lube distillate yield and quality from LFRAC (distillation simulation software) using boiling point distribution curves generated from GCD data rather than 15/5 plus Hivac data; and (c) use of actual laboratory-derived 100N EXPARMS rather than values calculated from 150N/600N distillates.

Predictions of lube distillate yield and quality from LFRAC distillation simulation software were found to be more accurate when boiling point distribution curves had been generated by GCD data rather than by 15/5 plus Hivac distillations. This was determined from analysis of data sets with good heat and material balance obtained from pipestills at Baton Rouge, Singapore and Strathcona refineries.

HYDROPROCESSING

1. Wax Isomerization

1.1 EXXSYN 6 Production (World Mutual)

WISR Runs 7, 8 and 9 took place in 1993, producing about 4350 tonnes of high quality EXXSYN that surpassed the manufacturing requirements.

Run Date	7 February	8 May/June	9 Oct./Nov.	Target
VI	142.4	143.5	143.0	141.8 min.
Viscosity @ 100°C, cSt	5.84	5.85	5.79	5.85
Noack vol., wt%	7.8	7.4	7.1	8.2 max.
Pour Point, °C	-21	-21	-22	
Oxygenates, ppm	<500	<500	<500	<500 (by HPLC)

Nitrogen blanketing of the waxy isomerate tank (Tk 522) was implemented before Run 6 and has been the key to suppressing oxygenates in the product. Further precautions were taken after WISR Run 8 to prevent oxidation of the heel left in Tk 522 between runs. A new inlet line was installed on the tank above the existing line so that the heel could be drawn down and solidified without blocking the inlet. This strategy proved successful: oxygenates in the Run 9 product were very low.

In each run, feed oil content and cut width were tightly controlled to guarantee high VI, low volatility EXXSYN.

Run	7	8	9
Oil in wax, wt%	8.4	8.4	(8.4, up to 18.8%)
fBP, °C	591	591	588
Std Dev. (σ), °C	38.4	38.4	37.8

Feed wax with up to 18.8% entrained oil was processed during the final few days of Run 9. Though this component of the product had lower VI and higher volatility, it represented only 10% of the final tank volume, and overall high product quality was maintained. Run 10, scheduled for February, 1994, is expected to use a lower cost feed with about 20% entrained oil (typical of the "3-2-1" operation on KDSR) with σ less than 40°C.

Crude variations will be carefully monitored in future for possible impact on 600N distillate boiling range distribution and subsequent EXXSYN volatility. Run 9 feed was derived from Arabian Light-rich crude, resulting in a feed with lower final boiling point. From a WISR perspective, Arabian crudes may be preferred over North Sea crudes because higher furnace coil outlet temperature may be used to maintain high crude rates through the pipestill while achieving the desired final boiling point and standard deviation for WISR feed.

1.2 Higher R101 Temperature for VI Improvement

(World Mutual)

KF-840, the catalyst in R101 of the Fawley WISR unit, is capable of converting naphthalene rings into higher VI iso-paraffins. Pilot plant and WISR test data confirm that this ring opening capability can be used to increase the VI of WISR products. This is of particular interest for high oil content feeds.

Tests carried out at SRC on feeds with 17 and 23% oil in wax showed VI credits ranging from 3 to 6 points when hydrotreater temperature was increased 20°C. A subsequent WISR test following Run 8 on feed having 8.4% oil in wax, showed a net VI credit of 1.5 points when R101 temperature was increased from 346 to 365°C, and product dewaxed oils had excellent oxidation stability, similar to that of EXXSYN from earlier runs.

A further WISR test at the end of Run 9, using 18.8% oil in wax feed, confirmed the effect: VI was improved by 2.5 points when R101 temperature was increased by 19°C to 365°C. The test was carried out at feed rates near record levels (12.4 m³/h) with no hydraulic limitations being observed. Run-down product met manufacturing targets. Based on these results, high-oil feed in combination with higher R101 temperature is likely to be used in Run 10, subject to ERCA's assessment of the product oxidation stability. Research in 1994 will focus on establishing the limits to acceptable feed cut width and oil content, given higher temperature R101 conditions.

1.3 EXXSYN from WISR Run 5

(World Mutual)

The oxygenate level in EXXSYN from WISR Run 5 (produced prior to the installation of nitrogen blanketing of the waxy isomerate tank) was sufficiently high (~3000 ppm) to impair its performance in stringent tests such as the Petter W-1. Studies at SRC and ERCA showed that the oxygenates could be removed by mild hydrofinishing over KF-840 (the R101 catalyst), restoring good performance in the Petter W-1.

Subsequently, oxygenates were successfully removed by hydrofinishing WISR Run 5 EXXSYN in R101. Unfortunately, an extract contaminant was picked up in the temporary line connecting the dewaxed oil tank and R101, resulting in an unacceptable performance in the Petter W-1 test. The re-hydrofined EXXSYN was subsequently sent to the FCC unit.

Any future requirement to post-hydrofinish EXXSYN (which is unlikely now that the waxy isomerate tank is nitrogen blanketed) would require a short jump-over line to ensure a segregated, clean routing.

1.4 Stacked Bed Catalysis

(Exxon Exploratory)

The key to producing good slack wax isomerate properties is proper balance between the rates of paraffin isomerization and naphthene ring opening. A higher relative rate of ring opening to isomerization is desirable when high oil content (lower cost) feeds are used.

Pilot studies have shown that ring opening can be enhanced in the isomerization reactor by combining two catalysts, one selective for isomerization and the other for ring opening. Catalysts suited for isomerization have higher acidity than those selective for ring opening. Experiments were tailored to a possible WISR opportunity since the top bed of the 3-bed reactor in R102 is currently empty. Lower acidity catalysts were combined with the existing RTF-10 catalyst in a 1:2 ratio with the test catalyst at the feed entry of the reactor.

Product quality improvements were observed when using either a 0.5% F version of RTF-10 or Y_2O_3 -doped silica-alumina (0.3% Pt) in combination with RTF-10 (1% F). Using 600N slack wax feed containing ~20% entrained oil, product VI was 2 to 5 points higher than for 1% F RTF-10 alone. Further investigation of stacked beds for R102 is contingent on whether benefits can be established above those generated by using more severe conditions in R101.

1.5 Light Isomerates

(Exxon Exploratory)

New low viscosity versions of EXXSYN from WISR are desired for applications such as 5W grade engine oils, hydraulic oils, ATFs and shock absorber fluids. ERCA has expressed interest in 2 to 4 cSt basestocks which have excellent low temperature fluidity. Some potential applications also require good biodegradability.

The 1993 program was guided by findings from the SILQ program which showed the relationship between process conditions, basestock properties and low temperature performance. Products were made at SRC from both 150N and 600N slack waxes and also from a highly converted 600N slack wax generated in the WISR in a brief test following Run 8. As anticipated, the high conversion products had the lowest Brookfield viscosities (in an Imperial Oil ATF), with the best results being comparable to ATFs formulated with PAO (Brookfield viscosity <10,000 cPoise). While low viscosity basestocks from 600N slack wax

have attractive properties, high conversions are required, resulting in low yields and high overhead volume which exceeds design limits in WISR stripping tower T102. The 150N slack wax feed is therefore preferred for light basestock manufacture.

Feed	150N SW (3% oil in wax)		PAO
Conversion to 370°C-, wt%	22	43	---
Viscosity, cSt @ 100°C	3.69	3.63	3.71
VI	141	133	121
Brookfield Viscosity, cP @ -40°C (in ATF formulation)	14,400	6,600	3,600

Evaluation by ERCA of pilot-produced isomerates from 150N slack wax in key formulations is expected to determine the feed requirements and process conditions for a WISR test run in 1994.

1.6 Slack Wax Isomerization Process Model

(Exxon Exploratory)

A slack wax isomerization process model has been developed to provide research guidance for feed selection/preparation, process optimization and planning. The model is based on a statistically designed set of experiments using the catalyst and process configuration of the Fawley WISR unit. The model, in the form of an EXCEL spreadsheet, predicts the impact of feed properties (such as oil content, distillation cut width) and process conditions (temperature, space velocity) on product quality. The model also predicts the volumes of the feed and product streams for the various units.

The model consists of several modules designed to simulate the steps of the WISR operation. These steps can be divided into two types - chemical reactions (hydrotreating and isomerization reactors) and separations (stripping towers and dewaxing unit). Changes in the molecular and distillation characteristics are predicted at each stage of the process and correlations are used to relate these changes to physical properties (viscosity, Viscosity Index, Noack volatility, etc.).

Predicted molecular changes resulting from chemical transformations are based on a parallel first-order network of reactions that use the individual reaction rates and activation energies for paraffin, isoparaffin, naphthene and aromatic conversion. The kinetic parameters were determined by non-linear regression of the experimental data.

The dewaxing module, based on correlations developed from the experimental data, predicts the molecular changes that occur across dewaxing. The stripping module is not yet completed and requires further work to predict changes in physical properties at topping temperatures other than 370°C (the temperature used in the experimental design).

Research in 1994 will concentrate on development of the stripping module, improving correlations, validation of the model with plant data, using the model to identify new business opportunities, distribution of the model to appropriate customers and documentation of results.

1.7 Hydrocarbon Synthesis Wax Catalysis

(World Mutual)

Large quantities of HCS wax have been isomerized under high conversion conditions (50-65% 370°C-) in a high mass velocity unit using RTSX-14, the "commercial readiness" catalyst. The lube properties of the 370°C+ material will be evaluated and compared with that of high conversion product previously prepared using low mass velocity conditions, to quantify the effects of mass velocity on lube quality.

100N and 175N basestocks have been prepared from a 343°C+ atmospheric bottoms material prepared from hydroisomerized HCS wax under low mass velocity conditions using an exploratory ERDL catalyst, Pd on amorphous 3A base. The products are being blended in ATF, PCMO and industrial oil formulations for comparison with similar products made during an earlier study using RTSX-11 and RTSX-14 catalysts.

1.8 Hydrocarbon Synthesis Wax Upgrading

(Billed)

An HCS wax upgrading program was carried out in SRC's high mass velocity pilot units to produce large volumes of fuel and lube products for future quality and end-use assessment. In all, 15 drums of light oil and 25 drums of wax feeds were hydrotreated and/or hydroisomerized. The impact of the light oil 260°C+ cut on meeting jet freeze quality was quantified. Undercutting the jet fuel product (160 to 246°C vs 160 to 260°C) eliminated the negative impact of this light oil feed component.

2. Severe HYDROFINING

Severe HYDROFINING was thoroughly investigated for applications at Port Jerome and Baytown and found to be an effective, durable process for deep HDS and for VI and volatility improvement. Presulfided KF-840 continues to be the preferred commercially available catalyst, although KF-852 appears to offer comparable VI improvement (presumably by ring opening of naphthenes in the feed).

Basestocks from severe HYDROFINING have demonstrated excellent oxidation stability and have passed Ames tests for mutagenicity.

2.1 Port Jerome

(World Mutual)

White oil production involves the process sequence: extraction - HYDROFINING - dewaxing, followed by hydrotreatment and hydrogenation, with Primol 352 typically representing 40% of the extraction slate. The use of severe HYDROFINING in its production, for example by operating Port Jerome hydrofiners A and B in series, was proposed as a way to permit debottlenecking by under-extraction of the raffinate. Extensive pilot tests at SRC showed this was feasible.

Primol 352 distillate was under-extracted by 5 to 7 VI points, giving a raffinate with >10,000 wppm sulphur. HYDROFINING at conditions simulating the A/B series hydrofiner operation (335°C, 0.4 LHSV, 550 psi H₂, 500 SCF H₂/B) easily achieved the

white oil plant feed sulphur specification of <300 wppm throughout the test (>3000 hrs). Furthermore, this operation did not jeopardize other product targets. All other feeds to Port Jerome's hydrofiner were tested during the program, with all products meeting specifications. Engineering has estimated that severe HYDROFINING would permit a 10% extraction debottleneck at a cost of about 500 k\$ US, about 10% the cost of a grass roots facility. Implementation has been deferred because extraction capacity is not currently limiting Port Jerome operation.

2.2 Baytown

(World Mutual)

EUSA marketing believes that low volatility light neutrals will soon be needed in greater volume than can be achieved by conventional processing. SRC studies show that Baytown could adapt the two reactors of the Specialties Development Unit (SDU) to make a low volatility 150N basestock by severe HYDROFINING of a 250N raffinate followed by dewaxing and blending with a conventional 100N basestock.

A 200N product with VI 9 to 11 points higher than that of its feed was consistently produced at 360°C, 0.5 LHSV, 700 psi H₂, 2100 SCF H₂/B. Subsequent dewaxing followed by blending with 10 to 20% 100N, gave a product which met the 150N volatility requirement of 14% Noack. Interest in a 2Q94 plant test in the SDU at Baytown has been boosted by an excellent result in a Sequence IIIE gasoline engine test that incorporated a severely HYDROFINED basestock from an earlier (1991) SDU test (see section 3.1).

Baytown is also interested in severe HYDROFINING for extraction debottlenecking. Pilot results with an aged KF-840 catalyst (skimmed from the LHU-1 unit) have shown that severe HYDROFINING (330°C, 0.6 LHSV, 650 psi H₂ and 450 SCF H₂/B) of an under-extracted 75N raffinate (79% saturates) met Baytown's minimum product specification of 85% saturates. Product collected at these conditions will be fractionated and assessed to determine if it meets EUSA's strict Orchex quality specifications.

Analysis of the data from the previous 1991 SDU test revealed that at 430 B/SD feed rate, the liquid hourly mass velocity was only 800 lb/hr.ft², while the criteria for new designs is >3000 lb/hr.ft². As well, investigation with the help of Baytown revealed that the SDU has an unusual bed distributor design which might be expected to give performance similar to the older sieve-chimney designs. Recent work with fuels hydroprocessing units has shown large credits when sieve-chimney distributors are replaced with more modern designs, particularly for low mass velocity operation. It is likely that less than optimum liquid mass velocity and distribution caused poor catalyst contacting efficiency in the last SDU test, debiting performance. Baytown has been advised that SDU performance in future severe HYDROFINING service can be significantly improved by the installation of new spray nozzle distributors and piping of the two reactors to run in series to double the liquid mass velocity.

2.3 Fawley

(World Mutual)

If WISR remains under-utilized, Fawley could make use of the R101 hydrotreater for severe HYDROFINING service at 1000 psi H₂ and up to 400°C. To demonstrate this potential, a 103 VI Fawley 150N raffinate was severely hydrofined over KF-840 in a high

mass velocity pilot unit at 335 and 365°C and other conditions typical of R101 operation for EXXSYN. Initial results show that super EBLA-type basestock (116 VI) was produced. Follow-up quality evaluation tests are underway at ERCA.

2.4 New Catalysts

(Exxon Exploratory)

Three variations of a KF-840-type catalyst with phosphorus content ranging from 0.4 to 4.0% have been prepared at ERDL and tested at SRC. All catalysts, including one with nominally the same amount of phosphorus as commercial KF-840 (2.8%), showed superior performance in severe HYDROFINING applications. For example, VI credits of 5 to 10 points were observed versus commercial KF-840 for a given set of conditions using a Baytown 250N raffinate feed. Detailed catalyst characterization studies are underway to establish which feature(s), besides phosphorus content, distinguish these catalysts. Continuing good performance in extended pilot studies will prompt discussions with a catalyst vendor, likely Akzo, to produce a semi-commercial batch of catalyst.

Model naphthalene, aromatic and paraffin compounds with carbon numbers in the lube boiling range have been prepared/purchased for a detailed investigation of the mechanisms underlying severe HYDROFINING. 1994 studies will be jointly conducted with Corporate Research.

3. Approvals

3.1 Basestock from Severe HYDROFINING

(World Mutual)

A severely hydrofined low sulphur product generated in a 1991 test run in the Baytown Specialty Development Unit has passed a Sequence IIIE gasoline engine oil test (which measures high temperature oil oxidation and wear control) in an ECI 5W30 SH quality formulation. The basestock, fractionated to 4.26 cSt @ 100°C from the total product, had 111 VI, 85% saturates and only 2.7 wppm sulfur. The excellent result of 82 hours for 375% viscosity increase (40°C) versus the minimum specification of 64 hours, clearly demonstrates that low sulphur base oils prepared by severe HYDROFINING can meet basestock approval requirements with current adpacks.

3.2 Jubail Hydrocrackate

(World Mutual)

Jubail hydrocrackate from Saudi Arabia is being considered as a supplement to A960 crude at Augusta and consequently has been submitted for toxicity testing. The results confirm that unextracted 100% hydrocrackate can be mutagenic. Extraction of 150N distillates containing either a 50:50 blend of A960 and the hydrocrackate or 100% hydrocrackate significantly reduced the mutagenic index. That for the blended baseoil was slightly higher than the index of the 100% hydrocrackate, suggesting that the extraction severity required to achieve a non-toxic baseoil may have to be modified when hydrocrackates are blended, rather than run as blocked whole feeds.

3.3 Crude Approvals

(World Mutual)

The nitrogen content of new North Sea crudes is approaching or exceeding ECI's self-imposed limits for acceptability. This could constrain crude choices for Fawley and Port Jerome. For example, 150N and 600N baseoils from Ekofisk crude, when produced according to assaying guidelines (to achieve minimum VI in this case), have nitrogen contents of 79 (vs 55 wppm spec.) and 159 wppm (vs 120 wppm spec.). Accordingly, Ekofisk derived basestocks have been submitted to lengthy D943 tests at ERCA to determine whether these nitrogen levels promote unacceptable oxidation. Since higher nitrogen levels are perceived as a continuing trend, the effect of HYDROFINING severity on final basestock nitrogen content is now being considered as part of the assaying sequence for higher nitrogen crudes. Preliminary calculations indicate that hydrofiners at both Fawley (the raffiner) and Port Jerome (the LOH units A & B) are capable of meeting final nitrogen requirements on Hudson crude, for example.

4. SILO

4.1 Characterization

(Exxon Exploratory)

The 1993 exploratory effort was directed at initiatives identified by the SILQ ("Science in Lubes Quality Action Team") program. A variety of techniques were applied to give insight, at a molecular level, into basestock properties such as oxidation behaviour, viscosity, VI and low temperature fluidity. Techniques are now in use to measure viscosity, cloud/pour point, density, miscibility, solubility and oxygenate content of very small (~0.1 ml) samples of lube basestocks.

An NMR method, developed at CR to understand low temperature fluidity, has provided some surprising insights. Diffusion coefficients of basestock molecules were measured as a function of temperature, and unexpectedly, there was little change in diffusion behaviour near and below the cloud/pour points. Since the NMR probe/signal accounts for >95% of the molecules, the results infer that just 5% of the molecules constrain the cloud and pour points. A further surprise was that isomerate molecules diffuse faster than PAO molecules at low temperatures even though formulated PAO basestocks are known to have superior low temperature fluidity. This suggests that manipulation, or removal, of a small fraction of EXXSYN might significantly improve product low temperature fluidity without compromising other parameters like VI.

Thermal diffusion experiments, performed at BSP, were used extensively in 1993 to discriminate different species in basestocks. EXXSYN 6 samples from WISR Runs 1, 3 and 5 were separated by thermal diffusion into ten 2 ml samples (ports 1 to 10). Sample VIs ranged from 172 to 95 respectively. Determination of sample oxygenate content and molecular composition showed that naphthenes, found in samples 9 and 10, are primarily responsible for baseoil oxidation. Blending of samples from ports 1 to 8 and 1 to 9 gave products having VIs of 155 and 149 respectively, and pour points of -20°C, similar to the whole EXXSYN sample (-21°C). These results again illustrate how basestock properties can be highly leveraged by a small fraction of the whole.

In a similar way, the effects of additives on basestock performance may be very focused. Accordingly, a new initiative involving the University of N. Carolina, is underway to measure basestock-additive interactions using a special NMR probe. Formulated basestocks are being studied, including those prepared from samples produced by thermal diffusion, at temperatures and shear conditions which simulate working environments. Initial studies are focused on measuring the effect of added C24 n-paraffin on PAO fluidity under high shear conditions and at temperatures near the pour point of the fluid. Follow-up studies will look at the effects of pour depressants.

4.2. Molecular Modeling

(Exxon Exploratory)

Molecular modelling calculations using Biosym software (in conjunction with CR) have been performed on pure and mixed hydrocarbons to clarify the molecular structure types and interactions that affect the low temperature fluidity of lube oils. Preliminary results have shown that mixtures of n-paraffins, at temperatures below the pour point, are aligned and close packed along the long axis of the paraffins, as expected. Calculations also show that, by contrast, PAO molecules exist in random arrays. Modelling of mixtures of iso-paraffins of the type found in EXXSYN is expected to identify preferred branching arrangements and combinations of arrangements that may be realistically produced by isomerization of slack waxes.

To complement the bulk fluid modelling effort, new software has been purchased to model interactions between hydrocarbons and catalysts, including microporous and amorphous (RTF-10 type) catalysts. It may be possible to predict product slate variations imposed by diffusional limitations within the catalyst structure. The acidity of catalysts is also being modelled to predict effects on, for example, isomerization branching density.

4.3 Oil Colour Measurement Using Chroma Meter

(Exxon Exploratory)

A Chroma meter CT-320, specifically designed for color measurement of petroleum products, is on order from Minolta Corp. This colorimeter can report oil/wax color as Saybolt (ASTM D156) and ASTM D1500 color numbers, eliminating the subjective interpretation of color measurement encountered with conventional Saybolt and ASTM D1500 scales. The measurement of petroleum color using the CT-320 colorimeter is expected to be published as an ASTM Standard method. Its use will be developed as a QC method under ISO 9000 qualifications.

A preliminary study indicated that white oil purity and haze could be measured using other "Color Systems" available in the CT-320. The use of such an instrument may eliminate the subjective interpretation of wax/oil color and haze in future.

5. Processes for Wax-Free Products

5.1 BP/UOP Catalytic Dewaxing

(World Mutual)

The program to develop a catalytic process for making electrical oils at the Baytown SDU has been completed. Changes in the naphthenics market, for instance a

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The program to develop a catalytic process for making electrical oils at the Baytown SDU has been completed. Changes in the naphthenics market, for instance a

growing customer preference for Type II electrical oils which permit higher product pour points, now make it unlikely that this project will proceed.

Studies in 1993 focused on activity maintenance issues. Run lengths of one year or greater are predicted for the standard conditions related to the early stages of the proposed project. However results at both BSP and SRC on South Louisiana 60N distillate suggest that doubling the initial feed rate, as proposed for a later stage of the project, would cause reactor temperatures to exceed the maximum before 9 months of operation. Thus, an electrical oil product comparable to current Univolt can be produced providing feed rates are kept somewhere below the levels proposed for the later stage.

5.2 Isomerization/Dewaxing with TON Catalyst

(Exxon Exploratory)

A major thrust of the exploratory program in 1993 was to identify catalysts and processes to make basestocks with excellent formulated low temperature fluidity. One approach is to hydroprocess at conditions which remove all of the residual wax from converted raffinates or isomerized slack waxes. For amorphous catalysts such as RTF-10, this can occur but at the expense of a large fraction of feed being converted to fuels. Attention therefore turned to certain microporous materials such as zeolites which are known to remove wax selectively. In particular, some outstanding results have been achieved with combinations of amorphous and microporous catalysts.

Zeolite theta, also called TON, was identified in 1992 as a microporous catalyst with excellent capability to trim dewax NCLs. Its capabilities are now known to be much broader. Raffinates and slack waxes were converted into low pour point products at moderate conditions (1000 psi H₂). The most significant results occurred using a 600N raffinate feed in a staged process simulating WISR-type configuration and conditions. The feed was first hydrotreated to remove polars then passed over a catalyst comprising particles made from a blend of RTF-10 and TON powders in a 3:1 ratio. Through both stages, feed pour point was lowered from +55°C to -35°C and VI was increased from 95 (dewaxed oil value) to 106, with only about 30% of the feed converted to 370°C- material. Furthermore, the low temperature performance of the formulated basestock was excellent.

The strategy of blending microporous powders with a powdered isomerization-type catalyst may help Exxon establish a proprietary position in this crowded art. The counter example, using separate particles of pure RTF-10 and TON in a 3:1 ratio is less effective. Alternatives to TON are being actively sought with help from a joint SRC/CR/ERDL/EE Lubes Catalyst Team since TON rights are believed to be dominated by BP in Europe and Mobil in N. America. Preliminary results with a pillared clay developed by CR show some promise.

6. Bulk Catalyst Development

(Exxon Exploratory)

The NiMnMo oxide bulk catalyst has been tested in a variety of applications from severe HYDROFINING (600 psi H₂) to the R1 catalyst of a fuels hydrocracker (2000 psi H₂). In most cases, the bulk catalyst activity was superior to that of conventional supported catalysts for ring opening of naphthenes to iso-paraffins.

The bulk catalyst also looks promising as an R101 catalyst for WISR. Using a slack wax feed containing 23% oil in wax, hydrotreated product produced using standard WISR conditions (346°C), had the same 2+ ring naphthene content as does current R101 WISR product from a feed containing only 8% oil in wax. Preliminary analysis also indicates the bulk catalyst to be far more effective for HDN and HDS of a VGO feed than C-411, the current choice of R1 catalyst in the Sarnia fuels hydrocracker.

A small commercial-scale production of the bulk catalyst by Calsicat has been commissioned to identify specific commercial opportunities.

7. Lubes from Fuels Hydrocracking

(World Mutual)

7.1 New Catalysts

A once-through fuels hydrocracking pilot plant run is underway to evaluate new catalysts for high quality lube production. A base case has been established with C-411 and IFP's HYC-642 as the R1 and R2 catalysts respectively. HYC-642 is considered a likely replacement for the current HC-8 at Sarnia. New catalysts include the bulk catalyst in R1, and a mixture of HYC-642 and Pd-loaded TON zeolite as the R2 hydrocracker catalyst.

7.2 New Feed

Lube distillates have previously been rejected as feeds to the Sarnia fuels hydrocracker (HCIS) because early correlations suggested their higher final boiling points would impair R1 catalyst activity (R1 run length is currently limiting). However, studies performed in 1991 and 1992 on Singapore distillates showed good catalyst life. A specific study of the C-411 R1 catalyst is in progress with Sarnia MCT-20 distillate. After 1 month, catalyst activity maintenance is excellent for hydrodenitrogenation. It is anticipated that Sarnia refinery will introduce MCT-20 distillate on a test basis in early 1994.

8. Hydroprocessing STAP and HYDROFINING Technology

During 1993 initial visits were made to Baytown, Wakayama and Singapore lube plants by the ERE and SRC lube hydroprocessing STAP team. In addition, a follow-up visit was made to Port Jerome, and hydroprocessing STAP participated in the Baton Rouge DEEP program. Contacts were also maintained with the other four lube plants.

Strathcona has replaced the catalyst in their lube hydrofiner with KF-742 and a new spray nozzle distributor has replaced their old version. The new distributor is the first commercial application of a new ER&E-proprietary design which gives more uniform feed distribution to the top of the bed. It is recommended for all new and retrofit applications where the benefits of improved catalyst utilization justify the replacement cost. Strathcona reports greatly improved hydrofiner performance, especially on MCT-60, a 1200N basestock which previously had been difficult to process to color specifications.

There appear to be opportunities to integrate HYDROFINING more effectively with distillation and extraction to optimize plant yields and producibility. For example, Wakayama may be able to increase HYDROFINING severity on 150N feeds to bring sulphur levels closer to 0.1%, down from 0.5%, while making products with acceptable oxidation stability. The associated viscosity decrease could be compensated for by cutting a heavier distillate, resulting in a significant yield increase.

A concern common to several sites is that many hydrofiner reactors are over 20 years old with outdated metallurgy and design features. Because of this, and safety considerations, many will have to be replaced in the next few years. The specifications which should be placed on future units need to be addressed in the near future, to ensure that future requirements are taken into consideration. Support for hydroprocessing STAP is growing and, in recognition of this, the program will be expanded in 1994.

9. Used Lube Oil Disposition

(World Mutual)

Disposal of used oil as refinery feedstock is of continuing interest. However, to avoid process and product quality upsets, some sort of pre-treatment will be required to remove gross contaminants, chlorinated solvents and at least some of the residual additives and wear metals. The pre-treatment step chosen must achieve the needed degree of clean-up while avoiding excessive capital or operating costs.

A variety of processes with varying degrees of complexity are available for purchase or licensing. Many of these have been evaluated over the past two years. The simplest processes, eg. the Shurtleff and the Aares, are high temperature atmospheric distillations which produce a distillate of cracked material. Much of the original chloride is present in the distillate, but the metals have been removed. Imperial Oil has been feeding small amounts of Shurtleff product to the Strathcona FCC fractionator since early 1993.

Several more sophisticated systems are available which involve multiple distillations, both atmospheric and vacuum. One example is the Enprotec Vaxon process, which gives very clean products and recovers most of the original lube basestock. Some processes even offer add-on technology to further reduce acid and chloride levels. However, such processes cost more and can only be economical if used to prepare higher value material, e.g. lube plant feed. Developments in used oil processing technology and changes in legal and environmental requirements in the different regions will continue to be monitored in 1994.

10. Madras Wax HYDROFINING

(Billed)

In 1993 two pilot plant programs were carried out in cooperation with ER&E Licensing for Madras Refineries Limited (MRL) of India. In the first, it was found that paraffinic waxes could easily be hydrofined to produce high quality waxes (FDA approval spec) at the conditions proposed by MRL. As these conditions (800 psi, 1 v/v, reactor temperature staging) are similar to present Baton Rouge and other licensee operations, this result was not surprising. The second program investigated whether microwax (from Bright Stock), could be hydrofined under the same conditions. Feed quality was poor with high

levels of asphaltenes and particulates being present, and catalyst deactivation occurred after just 48 hours on stream. At other locations, rapid catalyst deactivation is offset by blocking operation with paraffinic waxes, by producing cleaner feeds or by periodic unit washing with a clean fuels stream. As none of these options would be available at MRL, it was concluded that ER&E could not guarantee process operability, and therefore would not license microwax HYDROFINING to MRL.

11. White Oil Hydrogenation Catalysts

(Billed)

Ketjen EK 2 nickel hydrogenation catalyst is used by both Port Jerome and Wakayama to produce medicinal grade white oils. In early 1993, Akzo's EK 2 manufacturing facility was destroyed by fire, and it is reluctant to reconstruct the plant because of the high cost. Akzo has offered modified versions of EK 2 as replacement catalysts, but these would be produced by a third party (Criterion). Hydrogenation catalysts which are available from other suppliers have shown good activity in preliminary testing by Tonen and ESAF. Long term deactivation studies with them began in 1993 at SRC, and will continue in 1994 on both mutualized and billed bases.

PROCESS ENGINEERING

1. Lube Correlations Maintenance

(World Mutual)

The five year plan was updated and now includes the new Windows version of PC LASSY. Work has begun on this tool and Visual Basic is being used for the foundation of WINLASS. It is planned to have a version for release by the end of the first quarter of 1994. Plans are still in place to migrate the mainframe Nomad databases to a LAN based database. The present plan is to use Microsoft Access for the migration, with the final decision to be made after drawing conclusions from PCN and other computing projects. For the Yield chart program, we will continue to wait for the Windows version of Pro-II and will evaluate the possibility of customization to allow linking of Lubes existing modules.

The PC LASSY release is scheduled for distribution 1Q94. During the year changes were made to the database procedures to exclude distillation data for 55 crudes which were based on 15/5 plus Hivac data. Additional programs were added to the new Windows version of the Lubes Toolkit done in Visual Basic. This is included in the current PC LASSY release.

This year the customer Feedback Resolution System was converted from Excel to Microsoft Access giving us more flexibility for data input and reporting.

2. SILO Research Guidance

(World Mutual)

Research guidance for the NiMnMo bulk catalyst was provided in the context of the Port Jerome severe HYDROFINING study. The catalyst is expensive because of its high cost per pound and its high bulk density. Recommendations were made that this catalyst should be applied to high value-added applications where conventional catalysts are a technical stretch, and that research should be directed at bringing down catalyst manufacturing cost while preserving much of the catalyst's benefit.

A research guidance study on functionalized basestocks was conducted and presented at the 1993 SILQ meeting. The concept is to incorporate additive functionality into the basestock itself by reacting it with appropriate chemicals. The advantage would be to lower the cost of formulated lube products and/or to make higher quality products. The guidance study looked at the typical components in a 10W30 motor oil, the unit cost of the basestocks and additives, the cost per gallon of formulated product and the total annual cost of each in all Exxon basestocks sold for motor oils. This identified the incentive for functionalizing each basestock. Issues to be considered in the research program were also suggested.

3. Outside Technology Evaluation

3.1 Chevron Isodewaxing

(World Mutual)

Activities focused on the evaluation of Chevron Isodewaxing Technology.

Isodewaxing is a new lube dewaxing technology that Chevron claims is a significant advance over conventional catalytic dewaxing and solvent dewaxing. Significant quantities of wax are isomerized to high VI iso-paraffins in one step at high yield. The catalyst and process are tailored to increase isomerization and minimize cracking. A patent and literature search was conducted and a non-proprietary meeting was held with Chevron. This resulted in an understanding of Chevron's catalyst and process and how it has been applied.

The catalyst is believed to be SAPO-11 containing platinum or palladium. SAPOs (silicoaluminophosphates) are three-dimensional crystalline microporous materials with properties similar to zeolites. SAPO-11 provides the moderate acidity while Pt (or Pd) supplies the hydrogenation functionality required for isomerization. Keys to the catalyst's success are the proper acidity, the size and oval shape of the pores and the fact that they are parallel and not interconnected. The process is tailored to operate at lower temperatures/ lower space velocities where isomerization is favored over cracking. The process was designed for hydrocrackate feed which is low in nitrogen (<10 wppm). It is not currently applicable to solvent-extracted raffinates without pre-hydrotreatment to remove nitrogen.

The process was commercialized at Chevron's Richmond, California lube plant with start-up in August 1993. The existing lube hydrocrackers provide feed to the Isodewaxing units. Chevron replaced the catalytic dewaxing catalyst in their catalytic dewaxer with Isodewaxing catalyst and likely built a new Isodewaxing reactor upstream of their existing heavy hydrofinisher. They shut down the solvent dewaxer that had been used to process 500N feed. The technology was used to increase the capacity of the Richmond plant from 9,400 to 10,900 BPCD (16%). Chevron was able to operate the existing hydrocrackers at lower severity (higher lube yield) and use Isodewaxing to boost VI to meet the same product specifications they were meeting previously. They are not using the technology to make higher VI lubes, although it has this capability.

Based on published Chevron information and non-proprietary discussions, much detail was developed about the Chevron Richmond operation including: crude feeds, typical hydrocracker feed and product inspections, detailed yields and yield charts for the plant before and after Isodewaxing was added.

Chevron is licensing the technology. Plans are to evaluate the Chevron catalyst for Exxon applications beginning 1Q94.

3.2 Mobil Isomerization Process

(World Mutual)

Mobil isomerization technology was also evaluated. Mobil was developing an isomerization technology while Exxon was developing EXXSYN technology. When the Fawley plant was announced, Mobil decided not to proceed with a plant of their own. Their concept is a two-stage hydrocracking-isomerization process for upgrading slack wax containing various levels of aromatics. Slack wax is upgraded to high VI lubes with limited requirement for product dewaxing. However, unlike Chevron's Isodewaxing process, dewaxing is still needed in the Mobil process.

Mobil's first stage carries out both aromatics saturation and ring opening under hydrocracking conditions (pressure >1500 psi, 5000 to 10,000 SCF/B, 343-400°C) using a typical Ni/W lube hydrocracking catalyst. The isomerization stage uses low acidity, large pore zeolite beta loaded with platinum, and operates at 290-370°C. The extent of dewaxing is not large. Dewaxing yields are 80-85% compared to 70% at Fawley WISR. However, dewaxing cost is more related to volume of material processed rather than volume of wax removed so this is not of great advantage. Overall yields and VI are similar to WISR but the process operates at much higher pressure. It was concluded that Mobil's isomerization process is not a step-out.

DISTILLATION

1. Distillation R&D

(World Mutual)

A study which ranks crudes according to their yield of light lube oil was published for use by affiliate supply personnel as a "quick reference" guide in making lube crude supply decisions. The best yields are obtained for those crudes which have the highest mid boiling point for a given viscosity.

A heat balance calculation for lube piperstills was incorporated into an Excel spreadsheet and made available to affiliate technical personnel to assist in troubleshooting.

Inferential control of the mid-boiling point of lube distillates is being investigated using the Sarnia lube vacuum piperstill. The mid-boiling point can be predicted using three operating variables. Improved correlations are being evaluated using more complex regression techniques and Exxon's neural net software.

The maximum yield of low viscosity basestocks, which are limited by volatility, can be obtained by a proper balancing of distillation and extraction. Studies of yield optimization were carried out for Strathcona, Baton Rouge and Sarnia. The study results were implemented in Strathcona in early 1993 and higher yields have been achieved. Recommendations for Baton Rouge and Sarnia are being finalized.

An SFC analyzer has been successfully tested in the Sarnia research labs as a tool for measuring the volatility of low viscosity distillates. The first application for this technology is the on-line control of MCT-5 volatility at Strathcona. The first phase of testing, carried out in the lab, demonstrated that the repeatability of the unit was well within Strathcona's requirements. The next phase of the lab program will involve continuous testing intended to simulate the operating conditions at Strathcona. The purpose of this work is to evaluate the ruggedness of the unit and to develop an operating procedure to deal with all aspects of the unit's operation before it is installed in Strathcona. In parallel with the lab work, the Instrument and Process Analytics Section of ER&E has prepared a Design Specification for the on-line installation at Strathcona.

2. Distillation STAP

(World Mutual)

The performance of PS-9 at Baton Rouge was reviewed following a turnaround. Poor performance of the wash bed of the atmospheric pipestill was identified and corrected. Testing of PS-8, as part of the DEEP study, identified future debottlenecking opportunities and laid the foundation for the PS-8 "sour-up" expansion project.

An engineering assessment of Baytown PS-3 was followed by a plant test which demonstrated the ability to economically switch between naphthenic crude and paraffinic crude and to make low volatility 100N distillate. Also, inadequate feed vaporization and poor vapour liquid contacting were identified as prime reasons for unstable operation of the Baytown SDU.

A new cutting scheme (150N/600N/CF) was introduced at Augusta VPS-2 to increase the yield of light lube distillate. Recommendations were made for engineering work to redesign the bottoms stripper liquid distributor on VPS-2 to correct low strip-out. STAP distillation has been working with Fawley Refinery to implement mid boiling point control on PV-3 and to minimize the backend tail of the heavy lube distillate. A review of proposals for deeper vacuum resid cut point at Port Jerome lube pipestill identified a potential concern for 600N distillate quality.

Following a STAP visit to Singapore, data analysis suggested that improved hardware would increase the recovery of light and heavy lube distillates. A new cutting scheme to increase distillate yield on crude was recommended by STAP distillation and is being reviewed by Singapore. Plant testing at Wakayama PVWJ-3 indicated that excessive leakage from Vac-1 sieve tray draw-off was occurring, and recommendations were made for unit modifications to eliminate the leakage.

A pre-shutdown review of Sarnia AVIS identified inadequate top pump-around circulation as a potential contributor to the plugging of the chimney pan. An additional draw-off was installed, fractionation efficiency was improved and increased yields of light lube distillate were achieved after the turnaround.

A plant test demonstrated the viability of producing 100N VI improver diluent oil for Paramins. The test complemented an IOL self-billed product development/ testing program.

A deterioration in the fractionation efficiency of Strathcona LVPs occurred in 2Q93. Statistically designed experiments were used to investigate the impact of the most important variables by carrying out plant tests. Improvements were achieved and follow-up is underway to correct hardware limitations.

EXTRACTION

1. Extraction R&D

(World Mutual)

Pilot tests have shown that injecting water into the extract solution releases an oil-rich stream which is similar in quality to the distillate. This can be recycled to the extractor to increase raffinate yield. A first commercial test of this technology was conducted at Baytown LXU-2 extraction unit during 100N operation. Injection of 1.5% water into the extract solution sprung 2.3 KB/day of recycle which increased raffinate yield by 1.8 LV%, absolute. In a distillate-limited environment, there are incentives of \$1 million per year to continuously practice recycle as demonstrated in the test at Baytown. The benefits at Baytown can be increased by improving the separation capability of the settling drum by the installation of crinkle wire mesh.

An inventory of Exxon and competitive lube extraction process and hardware technology has been developed to determine the best research strategy. The comprehensive survey included: 1) world patents since 1970, 2) ER&E and SRC competitive assessments and 3) Texaco Development Company reports acquired as a result of the Texaco/Imperial Oil merger. Exxon has freedom to operate at water levels above 1% in NMP which affords treat and yield credits over the Texaco "dry" system. This was a major element of the ER&E/Texaco Development agreement on NMP solvent, negotiated in 1979. Texaco has significant activity in improving yields using recycle techniques and reducing the volumes of off-spec products during stock switches through process control. Recent research at SRC on the use of temperature gradient, extra staging and recycle enables us to achieve over 90% of the theoretical yield on extraction. Exxon initiatives in analyzer and process control are underway to reduce losses due to stock switching. The thrust of extraction activities over the next few years is to focus on application of existing technology through STAP.

2. Extraction STAP

(World Mutual)

Augusta lube extraction units have experienced one or more unplanned shutdowns per year due to fouling of the extract recovery towers. A joint EE/EEL/SRC task force was established to identify the cause of the problem and to recommend appropriate unit modifications. The foulant, which deposits in the chimney pan and upper stripping section, was found to be mainly iron sulphide formed by high temperature sulphur corrosion in an acidic environment. The source of sulphur is the fuel gas used for stripping, which contains 200 to 500 wppm H₂S. The task force visited Augusta and made the following

A plant test demonstrated the viability of producing 100N VI improver diluent oil for Paramins. The test complemented an IOL self-billed product development/ testing program.

A deterioration in the fractionation efficiency of Strathcona LVPS occurred in 2Q93. Statistically designed experiments were used to investigate the impact of the most important variables by carrying out plant tests. Improvements were achieved and follow-up is underway to correct hardware limitations.

EXTRACTION

1. Extraction R&D

(World Mutual)

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recommendations: 1) convert stripping gas to nitrogen, 2) filter the entire chimney pan liquid to remove solids, 3) utilize the existing ZnO sulphur trap to remove any incremental reactive sulphur. Augusta plan to implement these recommendations in 1994.

An ion exchange bed for removal of acidic components from NMP has operated continuously at Strathcona for about seven months. The total acid number of the circulating NMP has been reduced by about 90% during this period. A number of issues including resin activity maintenance, resin capacity and regeneration efficiency must be resolved before this technology is available for general commercial application.

An analyzer to predict dewaxed oil VI from spectral analysis of waxy raffinate has been under development with Corporate Research. Correlations have been developed which permit prediction of VI within ASTM reproducibility limits for 100N and 600N dewaxed oils. The analyzer will be plant tested at Baytown in 1Q94.

Plant performance tests at Baytown LXU1 evaluated throughput limitations and provided a basis for further engineering studies for capacity improvement.

A STAP extraction visit to Singapore provided recommendations to improve yield, improve the control of the recovery tower and the water in solvent and reduce variability in laboratory results.

Extraction plant tests were carried out at Baton Rouge as part of the DEEP performance testing. This program identified an immediate opportunity for increased raffinate yield, and provides the basis for defining the logical sequence of debottlenecking steps and the role of new technology to increase base oil producibility.

3. Extraction Solvents

(Exxon Exploratory)

New extraction solvents to replace N-methyl pyrrolidone (NMP) and Phenol were investigated. An extensive literature search was done to identify potential lube extraction solvents based on their physical properties (i.e. specific gravity and boiling point) and structure (heteroatom containing ringed compounds). Out of a database of 175,000 chemicals, only 100 had the required physical and molecular properties. Of these, many could be eliminated as solvents because of chemical instability or availability issues. Several extraction solvents were evaluated using a new screening technique involving the batch extraction of a mixture of synthetic lube oil molecules consisting of saturates, 1-ring, 2-ring and 3-ring aromatics. Only one of the solvents tested, tetramethylene sulfoxide, was identified as being marginally better than NMP/water. Patent coverage is being pursued. The main advantage of NMP as an extraction solvent is its high capacity, which can be traded-off for increased selectivity by the addition of water. This gives NMP a degree of processing flexibility that most other potential solvents lack. No further research in this area is planned for 1994.

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KETONE DEWAXING

1. Dewaxing Process Model

(World Mutual)

Correlations of data from the ketone dewaxers of all Exxon affiliates show that dewaxing filter rate and wax cake liquids/solids are strongly correlated with the raffinate boiling point distribution (mean, variance, skewness and kurtosis) as well as the raffinate wax content. In early 1993, these effects were confirmed at Baton Rouge and correlations were developed which were specific to the KDLA. Based on these learnings, pipestill operations were altered to produce significant reductions in the variance of the boiling point distributions of all distillates. These changes produced significant yield and throughput credits in dewaxing.

Statistically designed pilot plant studies were used to identify key variables in the dewaxing of Fawley 600N by incremental dilution. These studies produced models which indicate the potential for a significant feed filter rate increase (>10%) on the Fawley 600N operation by making changes to the chilling profile and solvent increments. Improved feed distillation was shown to give an additional significant increase in feed filter rate.

2. Dewaxing STAP

(World Mutual)

As part of the "DEEP" STAP effort at Baton Rouge, statistically designed plant tests were used to model the dewaxing performance of three major feeds at Baton Rouge: Paraffin Distillate, LP100N and NL500N. The key variables selected for study were dilution ratio and the injection rate of Baytown slack wax. For each set of conditions studied, the KDLA was operated at maximum throughput. Linear programs were then applied to these models to select those conditions which maximized saleable product and yield on raffinate. These optimized conditions resulted in increased throughputs of +2500 B/day (+12%) on Paraffin Distillate, +1300 B/day (+6%) on LP100N, and +6100 B/day (+47%) on NL500N.

A STAP study on the Sarnia dewaxer in November demonstrated opportunities for increasing the dewaxed oil yield of MXT 5 by 2 to 3% by operating to a lower target for second stage oil content and increasing the pour point target to -18°C (max) from the present -21°C. The potential for energy savings due to decreasing the hot wash frequency by 30 to 50% on the MXT 5 and other light stocks was highlighted.

Technical assistance was provided to Sarnia to help identify the causes of a serious MCT 60 haze problem, and advice on filter re-clothing was given to help to correct the problem.

At Strathcona a training course for dewaxer operators was conducted in November. In addition, Strathcona converted one scraped surface chiller (SE-604) to Borsig internals during their shutdown in August. Performance evaluation is now in progress to see if heat transfer and pressure drop credits can be realized.

3. Exploratory Dewaxing

(Exxon Exploratory)

The development of a model for wax crystal growth in the DILCHILL process by Professor Allan Myerson at Brooklyn Polytechnic continued. The objectives for 1993 were to incorporate into the model the effects of scraped surface chilling, and model verification using plant and pilot plant data. To meet these objectives, wax crystal size distributions were measured on DILCHILL outlet and scraped surface outlet slurry samples of SL150N and NL500N at Baton Rouge. Drum quantities of these feeds were dewaxed using the DILCHILL pilot plant facilities and wax crystal size distributions were determined on the pilot plant slurries.

Studies at CR indicate that coating of stainless steel surfaces with a monomolecular layer of n-C₁₈ silane may prevent wax adhesion to a cooled surface. Procedures have been developed by CR to apply this coating to a stainless steel heat exchanger. Pilot tests are planned at Sarnia for early 1994.

At the University of British Columbia, Professors Watkinson and Epstein have developed methods to measure wax fouling resistance on cooled surfaces. This capability will facilitate the development and screening of the effects of surface modification on heat transfer performance when cooling wax-solvent slurries.

Attempts to duplicate in the laboratory the "ART" process as described in international patent application PCT/US92/00666 using a typical 150N raffinate were not successful. The largest pour point reduction obtained in lab experiments was 16°C with two stages of dewaxing and some external chilling required. No further work is planned.

The effect of basestock composition on low temperature performance is a key parameter. Consistent with this, a study has been carried out to investigate the effect of dewaxer feed composition on dewaxing performance. Normal paraffins are expected to have the greatest impact on dewaxing, because of their high melting points and low solubility. A two variable statistically designed pilot study investigated the relationship between feed wax and n-paraffin content, and feed filter rate and liquids to solids ratio for a 600N dewaxer feed. Within the variable ranges investigated, dewaxing performance was found to be strongly affected by the feed wax content, but n-paraffin content had no effect.

4. Membrane Recovery of Dewaxing Aid

(World Mutual, Exxon Exploratory)

Pilot testing of polyarylsulfone (PAS) membrane elements demonstrated a tendency for feed bypassing due to weakening of an epoxy/aluminum interface during thermal cycling. An improved 8" element design was implemented which eliminated this problem. Pilot testing using modified elements showed that high fluxes (500+ L/m².day for 600N slack wax feed, 1 wt% active aid) and excellent selectivity were obtained, and element integrity appeared to be excellent. Flux decline was not observed except during operation at high aid concentrations (>3 wt%), and flux models were developed for 150N and 600N feeds using the flux data obtained in pilot plant testing.

Pilot testing under conditions simulating potential upsets in the Singapore Refinery unit (eg. high aid concentration spikes and hot and cold unit shutdowns) showed that the element package is very robust. Membrane performance could be rejuvenated following testing with 1 wt%, but not 3 wt% active aid in 600N slack wax, by operation on light oil.

Testing at Singapore refinery confirmed the robustness of the element package. However, using 10 micron pre-filtration, flux decline was observed which is believed to be due to membrane fouling by species in the refinery feed (solid particulates and/or asphaltenes). A test using 10 and 1 micron pre-filters in series to remove most of the foulants was carried out, and flux decline was again observed, but at a reduced rate.

PROPANE DEWAXING

1. Propane Dewaxing STAP

(World Mutual)

At Augusta, Viscoplex dewaxing aid was tested as a replacement for ECA 9555/Plexol for 600N and Bright Stock. Viscoplex was found to be unattractive for Augusta at this time because it reduces feed rate on Bright Stock.

A capacity test was carried out on the propane dewaxing unit at Baytown. Higher chiller levels and lower cold dilution ratios were used to decrease the chilling rate and thus increase plant rates to a record level of 9600 B/day on 600N.

The Propane Processes Workshop was held at Singapore in 1993 with participants from all propane dewaxing and deasphalting affiliate sites, as well as ER&E and SRC. The workshop provided a forum for the transfer of improvement ideas between plants.

During the Wakayama STAP visit, an increase in yield due to increased 1st stage wash was demonstrated. In addition, several minor equipment modifications were recommended, which were estimated to result in an approximate 10% increase in feed rate. Very poor second stage filtration was observed due to wax crystal damage between the first and second stages. The crystal damage could be reduced by reducing pressure drop across the exchanger and replacing the first stage wax pumps. An opportunity was identified to reduce the dewaxing aid dose by replacing prechillers. This was the first time that minifilter data was obtained on warm-up-deoiled slurries to aid in the plant analysis.

A Singapore STAP visit demonstrated a viable 2-stage dewaxing operation on 150N and identified several items that would increase the maximum dewaxed oil production. Singapore is now using the full 2-stage operation on 150N (second stage filtrate to first stage wash), giving record high yields and near-record DWO production. Improvement items include cleaning the wash headers inside the filters (mechanical cleaning or replacement) for yield increases of 1 to 4%, depending on the grade. In addition, the refrigeration compressor capacity may be increased if the speed of the machine can be increased about 10%, as was done successfully at Baytown.

DEASPHALTING

1. Deasphalting STAP

(World Mutual)

The STAP visit to Augusta verified the effectiveness of a new propane knockout drum for removing entrained asphaltenes from the circulating propane. The asphaltenes were measured by installing small filters on slipstreams of the propane before and after the drum and weighing the residue. Cleaner propane has resulted in better propane dewaxer performance.

Deasphalted oil was also found to be entrained in the recovery tower overhead. As a result, Augusta experiences significant fouling of the air fin exchangers just downstream, which limits plant capacity. Recommendations have been made that the equipment be cleaned during the next turnaround and that facilities be added to allow on-line cleaning.

2. ROSE Fouling Study for Baytown DAU

(EUSA Billed)

Increased solvent treat rate has been shown to reduce the fouling tendency of deasphalted oil for several types of Baytown resid. This information can be used to evaluate the potential for reducing fouling in the Baytown Residual Oil Supercritical Exchanger (ROSE) exchangers by increasing solvent treat ratio.

OPERATIONS

AUTOMOTIVE TEST SECTION (ATS)

1. ENGINE TEST LABORATORY (ETL)

In addition to the maintenance of ongoing operations, three new test stands were commissioned in 1993.

(1) Intake Valve Deposit (IVD) Test

This test, using the Ford 2.3L twin spark plug engine, was commissioned early in the year. Precision, repeatability and discrimination were demonstrated. A series of over 20 tests was conducted for the Fuels Products Section for the optimization of gasoline detergency treat rates, as well as evaluation of pipelined Kamloops gasoline. Four tests for Exxon Chemicals, Paramins were also conducted. This test replaces the higher cost, less precise BMW 318i IVD industry test.

(2) GM 6.2L Roller Follower Wear Test

This proposed industry standardized test is one of the required tests for the next generation HD engine oil category (PC-6). Commissioned in mid-year, two reference oils were tested as part of the industry test development efforts. Lubricants (EDTL) Section has been using this stand for exploratory research and formulation evaluation.

(3) HD Fuel Economy Engine Oil Test

This test stand, commissioned in late 1993, uses a Detroit Diesel Corp. Series 60 engine. A proprietary procedure allows the measurement of energy conserving performance of HD engine oils. Elaborate controls and accurate measurement, coupled with a test procedure designed to simulate various operating conditions, are factors enabling the test to meet precision requirements.

The planning of one new test stand, the Sequence VI-A, which replaces the industry Sequence VI fuel economy engine oil test, is under way. Commissioning is expected to be early 1994. Other active test types in the ETL included DD6V92TA heavy duty engine oil test, Caterpillar 3304 natural gas engine oil test, Sequence VI fuel economy engine oil test, Caterpillar single cylinder copper corrosion test, and AVL/Cummins HD engine oil test.

2. ALL WEATHER CHASSIS DYNAMOMETER (AWCD)

Due to the light demand from internal customers in 1993, significant testing, primarily at low temperatures, was done for the Ford Motor Co., Dearborn, MI.

Internal programs included an investigation of a low temperature gel problem in passenger car engine oils. A low temperature engine oil pumpability test program was carried out in 1993 and will be continued in 1994. Both programs are sponsored by ERTL.

PROCESS AND TECHNICAL SERVICES

The Process and Technical Services section continued to provide high quality technical support during 1993. The P&TS personnel are responsible for operating our world class pilot plant facility, providing maintenance and design services in the mechanical, instrument and electrical areas, and supplying engineering/contracting support for a variety of research projects.

Pilot plant operations demand increased by approximately 5% during 1993 and averaged 10 "typical" hydrofiners.

Specific lube programs supported included: Wax Isomerization (WISR), Raffinate Hydroconversion, Severe HYDROFINING (Baytown), Hydrocracking Catalyst Studies, HCS Wax Isomerization, VI Improvement, White Oil Catalysts, Phenol Alkylation (Paramins).

Specific refining support and miscellaneous programs included: Hydrocracking Program (Benecia), Hydrocracker R2 Catalyst Evaluation (Sarnia), C3 Oligomerization (Chemicals), Project Orange, Extraction/Dewaxing Programs, Resid Ultrafiltration.

Several major mechanical projects were progressed: CFC Phaseout, Building Energy Management System, Pilot/Batch area automation projects, Sandbath reactors/intermediate feed tankage.

Team oriented initiatives contributed greatly to our continuous improvement program in 1993. Our loss control team and steam team were instrumental in improving our work processes/safety performance and substantially reducing energy consumption. In addition significant efforts are underway in the areas of Operations Integrity and statistical quality techniques. These efforts are aimed at improving the quality of our product at a reduced cost for our customers.

ANALYTICAL

In 1993, the Analytical business unit continued its drive to improve customer satisfaction with high value products and services. Overall, sample loads remained near historic levels (approx. 41,000), and there was a significant increase in developmental and exploratory work. With staffing 25% lower than two years ago, it has been possible to meet customer expectations only through significant work process re-design and flexible, highly integrated teams.

Analytical's main customer groups remained unchanged from 1993: 87% from SRC, 7% from billed work for IOL and Exxon, 5% from World Mutual R&D for conventional and next generation crude assays, and <1% from true external customers primarily in the Sarnia area.

During 1993, we completed 1992's drive to a fixed cost/test basis, which in almost every case remains lower than or equal to those of commercial labs. Our 21st annual Quality Assurance/Quality Control report noted the addition of several new tests and the confirmation of our capabilities in industry round robins. More than 83% of our tests had a Process Capability Cpk > 1.3.

To address contracting and ongoing communication with customers about their contracts, the implementation of the first phase of our Laboratory Information Management System (LIMS) occurred in 1993 (plasma wear metals, viscosities in conjunction with Customer Service Laboratory). When completed, customers will know the cost, turn around and quality prior to submitting a sample, and will be able to track and steward its progress in real-time. It will also aid Analytical staff in knowing and managing the total scope of their work in light of customer expectations. To date, LIMS has delivered its advertised large improvements in productivity and capability (commonly >25%), encouraging us to continue implementation in 1994. As well, it will be a key system for ISO 9001 accreditation and Operations Integrity.

1. Molecular Crude Assays

(IO Products; World Mutual)

Driven by the universal need of supply and processing facilities for rapid assessment of crude quality, new mass spectrometric (MS) techniques were developed. The most important and fundamental development was a 1.5 hour method for LV% tBP of a whole crude based on GCD/MS. After incorporation of innovative GC hardware and development of new molecular algorithms, this method is in the final development stages addressing accuracy (versus 15/5 and Hivac physical distillation of 30 varied crudes) and precision. Plans for running this as a routine service in 1994 were developed so that customers will get results electronically in 1-2 days.

Several rapid MS methods were developed for characterizing corrosive sulphur species in crude or refinery streams. Most important is a 10 minute direct measurement of Total Reactive Sulphur which is used by ER&E in refinery corrosion models, and which is currently only available from multiple wet chemical methods. Individual species (eg. sulphides, disulphides, thiophenes, total sulphur) can be determined by new MS correlative methods.

To address quantitative analysis for ppb levels of Ni, V, Fe and Na in crudes and refinery streams, a new method was developed which completes the analysis in minutes. This is based on an ultrasonic nebulizer coupled to a plasma spectrometer and will eliminate a large volume overnight wet ashing step.

2. General Technical Contributions

(a variety of Imperial & Exxon customers)

Using the molecular information obtained during the GC/MS research on LV% tBP of a crude, scoping studies have shown its ability to predict physical/chemical properties of fractions or blends of the crude. For example, pour points of selected fuel fractions have been accurately predicted *without* distilling the crude.

Due to the negative economic impact of product volatility specifications on yields of light 100N lube baseoils, the precision of GCD was significantly improved to permit better control of lube plants.

As part of the SRC Customer Support Lab project to improve used oil analysis services, enhancements were made to the measurement of wear metals by plasma spectroscopy (detection limits and accuracy extended for Mg/Ca/Zn/P/Ba, while also lowering costs) and methods were developed for a new infrared used oil analyzer.

As part of our responsibility to support the Sarnia polyethylene (PE) research/plant, new liquid chromatographic methods for additives analyses were developed (eg. butylated-hydroxy-toluene in PE) and existing ones improved. These are critical analyses for regulatory requirements (eg. polyethylene used as food containers).

Ongoing expertise and support were provided to IOL refinery/plant Quality Assurance lab operations. For example: (a) when Sarnia QA Lab's main data system failed, we ran critical analyses and aided with an emergency installation of a new system, (b) we developed a 10 minute method to determine 50-2000 ppm of diethanolamine and triethanolamine in Nanticoke water samples, and (c) improvements were suggested to QA operating procedures in several elemental analyses.

In response to increased customer demand (eg. IOR, Strathcona and Nanticoke refineries), conventional assay services were extended and improved. As well, an ER&E round robin of D-2892 atmospheric and D-5236 vacuum distillations confirmed our ability to produce boiling point data within ASTM's 2 sigma confidence ranges.

Furthering 1992's development of simulated distillation methods for a process analyzer supercritical fluid (SFC) instrument, we confirmed its suitability for on-line analysis of lube basestock volatility.

A new liquid chromatographic method, CR-ASL's Weak Cation Exchange (WCX), was successfully commissioned. It can characterize and quantitate polar compounds in samples such as lubes, heavy oils, crudes and asphalts. It is especially important for determination of polar oxygenates in non-conventional lubes such as EXXSYN.

In support of concerns about residual processing solvents in products, a gas chromatography method was developed to rapidly analyze for ppm to % levels of phenol and/or NMP in oil.

In support of formulated lubricant development, a thermogravimetric analysis method was developed for soot in used engine oils. As well, physical characterization methods for heat capacity and heat of vaporization have been developed. All three new methods were validated in round robin tests with other sites and companies.

An HPLC method, based on the existing Fast Lubes Separation (FLS) method, was developed to accurately determine < 1% aromatics or saturates in lube samples (important in research programs developing high VI basestocks).

Quantitative elemental analysis of water samples was made quicker and cheaper, and detection levels lowered 10-20 fold to ppb levels with the installation and development of methodology for using an ultrasonic nebulizer on existing plasma spectrometers.

The gas chromatographic simulated distillation (GCD) method for high boiling hydrocarbons (C10-C90, 174-692°C) was converted to capillary column technology to realize improvements in precision (2-6 fold) and reliability without sacrificing accuracy.

TECHNICAL INFORMATION CENTRE

The SRC Technical Information Centre maintains the principal collection of company downstream information in the form of books, manuals, journals and reports. During 1993, the Information Centre added 289 new titles to this collection and filled 507 requests for in-house journal articles, patents, reports and correspondence. 648 requests were processed for external material, books, patents, journal articles and conference papers. 115 literature searches were performed.

The Information Centre Team underwent several staff changes and has benefited by maximizing the opportunity for cross-training. This multi-trained team provides efficient service to our internal and external customers. Continuous enhancements have been added to the services provided by the Information Centre as a direct response to our clients' requests. These include remote access to the collection, on-line search capabilities, current awareness publication with abstracts on diskette and U.S. Chemical Patents on CD-ROM.

During 1993, 48 patent memos were written and 26 patents were issued to members of the Department. Sixty-one formal reports were issued. Titles and authors of granted patents and formal reports follow.

The Research Technical Information Centre maintains the Patent Honour Role Board that recognizes inventors with 10 or more issued patents. This year J.P. Boyle was recognized for his excellent achievement.

SPECIAL ITEMS

PRODUCT DEVELOPMENT PROCESS & THE RESEARCH PROJECT MANAGEMENT SYSTEM

In 1993, the Lubes and Specialties Business Unit of IOL Products Division adopted a four step model for the Product Development Process (PDP). The four stages are:

1. Preliminary Assessment: A proposed product development is initially screened for strategic fit, cost/benefit, chance of success. A go/no go decision is made with feedback to the originator.
2. Detailed Assessment: Business and technical targets are more clearly defined for projects advancing from Stage 1.
3. Development: Product sourcing (buy vs make) decisions are made, and supply and market plans developed for projects passing Stage 2. From a research point of view, the greatest effort will occur when a decision is made to formulate in-house.
4. Product Launch: This is the commercialization phase where plans are executed, product made available, and assessment is made of plans vs actual, delivery of benefits, costs and targets.

Within the Products Division of SRC, the concepts of the PDP have been applied to the creation of a project management system which is intended to allow measurement of progress of those product developments in which Research plays a role. This management system consists of four elements:

1. The Product Development Process - is used as the conceptual basis throughout the system.
2. Technical Planning Groups - The agendas and outputs of the TPG meetings are organized to deal with research projects within the context of the PDP.
3. Products Project Manager - This is a computer data base of IOL research projects. Projects are registered on entering Stage 2 of the PDP and objectives, targets, budgets, business prize, milestones, deliverable schedules are identified and updated.
4. Program Area Summary - Projects are summarized on a monthly basis to allow stewardship against the time and dollar commitments for each project within the area.

The Product Development Process and the project management system are key to measurement, analysis and improvement of the Products Division's commitment to provide timely, valuable information to our internal customers. Application of these processes will also facilitate achievement and maintenance of ISO 9001 accreditation for the Divisions' Sections.

TOTAL LOSS CONTROL INITIATIVES

In the continuing drive to reduce costs arising from all types of incidents, new initiatives were implemented by Operations business units to address Total Loss Control. While a prime focus remains on safety, the programs have been designed to capture information on all forms of loss, including those in areas such as quality, unnecessary rework, etc.

The programs have several notable features, including an environment which encourages the capturing of all events, new tools customized to the job, processes which facilitate the timely resolution of identified incidents, monthly tours employing safety experts, and the development of higher level learnings which address root causes. This input is key to SRC's Operations Integrity and ISO accreditation initiatives.

OPERATIONS INTEGRITY IMPLEMENTATION

The department continued its program of implementing the IO Products Operations Integrity framework in 1993. OI systems are being developed to meet the expectations contained within the 11 Operations Integrity Elements.

Champions within Research developed a draft set of OI standards and implementation plans for the department in 1993. The Operations Integrity Steering Group and Research Leadership Team provided strategic guidance. The OI implementation team leader provided overall project management and direction. Implementation of some systems began in 1993 and full implementation is planned for 1994.

An assessment of the status of the current OI systems in SRC was carried out December 6-8, 1993. The assessment team talked with a wide cross-section of the department, made various field observations and reviewed relevant documents. The overall rating was 2.0, using the Imperial Oil Limited Products Division "Operations Integrity Measurement Tool and Self-Assessment Guidelines - July 1993" to assign the rating. The overall department goal for 1994 is to reach a rating of at least 3.0. The 1994 OI implementation task plan for SRC will be developed by mid-January 1994.

STATISTICAL CONSULTATION

In a continuing effort to improve the quality and efficiency of our scientific research programs, Engineering Services Canada has been contracted to provide statistical consultation services to researchers in the department. Personal consultation sessions are available on a monthly basis, and continuing support is available through computer linkup.

The major areas of interest to researchers are:

- Experimental Design - the disciplined approach to experimental planning, to ensure that information-rich data are collected efficiently.
- Analysis of data - the separation of the experimental signal from background noise.
- Statistical Process Control (SPC) - the development of adequate Quality Controls for the process under investigation as well as for sample analysis.
- Statistical Computation - the use of SAS or other statistical tools.

The consultation service also includes capability development for researchers. In 1993, a number of interactive workshops were held for the Lube Process Division. These were:

- An overview of PPMS (Plant Performance Monitoring System)
- DOE (Design of Experiments) and Basic Statistics
- Empirical Modelling using Regression Analysis and Neural Nets (overview of Automatic Process Control). This was presented jointly with Exxon Research and Engineering.

N.I.C.H.E. (New Ideas Capturing a Happy Environment)

The NICHE project was developed as a tool to assist Research Department members in achieving a supportive work environment. Input was first obtained from peer groups during informal discussion sessions, which lead to two initiatives: feedback on specific issues of general interest, and a re-designed formal communication framework.

The feedback on specific issues has been used to improve the general understanding and awareness of issues affecting the whole department, for example, compensation and the performance enhancement system. As well, some department members have participated in focus groups to further work-related ideas.

The re-design of the departmental formal communication system reduced the number of meetings to one per month for each division and increased the level of information exchange. Operations Integrity, Safety, Operations services and Human Resource

topics are evergreen items. The new format has reduced the time spent on one-way communication, increased employee contact with division managers and improved the consistency and timeliness of departmental communications.

SHARED RESEARCH PLANNING

Shared research planning is a new approach to the planning and funding of Products research programs of interest to more than one Exxon region. The system was developed during 1992 and initiated in 1993, and will replace the World Mutual products research planning system by 1994.

In the Spring of 1993, each region (IOL, EUSA, ECI) developed a prioritized list of potential 1994 products research projects. Business and Research representatives of each region then met to identify opportunities to "share" research projects of mutual interest. The proposed research plan resulting from those discussions was later reviewed with the Business Unit Managers and Lab Managers at the Program Planning Committee (PPC) Meeting. As a final check, the Research Strategy Committee (RSC), of which Bill Innes is a member, reviewed and endorsed the final proposal for 1994.

Efficiencies gained by sharing project or technology developments enable the regions to fund more research projects within their affordable budget levels. A key advantage over the previous World Mutual planning system is the intimate involvement of the business customers in the planning process. The new system also fosters the sharing of research projects between any two regions.

The mutualized exploratory projects research system also underwent significant change. Prior to April 1, 1993, all Products exploratory research was conducted at Products Research Division (PRD) in Linden. On April 1, the Exploratory Division was dissolved and the exploratory researchers moved, with their projects, to the fuels and lubes divisions at PRD. Approved 1994 Exploratory projects will be conducted at the most appropriate location, as decided by the Products Research Management Team (PRMT), of which Clarke Henry is a member. In 1994, a basestock exploratory project will be conducted in Sarnia.

Overall, the new "shared research planning" system has many advantages over its predecessor, and should continue to enhance the contribution of research towards business success.

CN TUNNEL

In early 1992, CN North America announced plans to construct a new rail tunnel beneath the St. Clair River from Sarnia, Ontario to Port Huron, Michigan. The 200 M\$ project is designed to accommodate double stacked container cars at higher speeds than is possible with the existing tunnel (completed in 1890). The new tunnel will be located 26 m north of the existing tunnel and is 8.4 m in diameter with a length of 1824 m, of which 600 m is below the river bed.

The routing of the tunnel takes it under the main Research building and various areas of Sarnia Refinery prior to reaching the St. Clair River. This location has several implications for ongoing operations of our Imperial Oil facilities.

Vibration levels and building settlement are the two major potential concerns. Specialists were consulted and facilities are in place to mitigate any vibration/settlement problems as the tunnel progresses.

Tunnel construction started November 1, 1993 and is expected to continue for nine months. Imperial Oil representatives are working closely with CN to minimize impacts on our operations.

SRC CUSTOMER SATISFACTION SURVEY BY CUSTOMER WINDOW

A customer satisfaction survey was carried out to better understand how SRC is meeting customers needs so as to improve SRC's contribution to the customer's success. The survey utilized the Customer Window technology, a method developed by Arbor Inc., licensed by IOL, and distributed within IOL by Elaine Garnett of Marketing. Elaine led a team of about thirty employees in a two day workshop that developed the strategy for conducting the survey. The survey was carried out by the Products Division, the General Manager Division, and the Lube Process Division. A questionnaire was developed to survey the customers on nine key product quality characteristics. The questionnaire contained two groups of 21 questions. In the first group, customers were asked to score the importance of 21 attributes (e.g. the cost of our service), and in the second group they were asked to score their degree of satisfaction with those same attributes. Four open-ended questions were included to capture literal comments and feedback.

Within the combined Products/General Manager Division, three customer segments were identified. A total of 194 survey were mailed and 125 received back, for an overall return rate of 64%. The numerical results were analyzed statistically. The literal data were used to confirm the conclusions from the numerical data, and to better define the actions required. A key area of interest to all customer segments was the time to complete and deliver research information. Some areas, such as reliability of information provided by SRC, value vs. cost, clarity of communications, innovativeness, and customer involvement in technology development, were indicated as very important by the customers. Any improvement SRC could achieve would result in a strong impact on customer satisfaction.

The Lube Process Division carried out a similar survey in collaboration with ERE, and obtained a 53% return rate from 32 questionnaire mailed. Reliability of information provided by SRC and clarity of communications were identified among the most important factors.

ISO 9001 REGISTRATION

ISO 9000 is an International Standard dealing with quality systems that can be used for external quality assurance purposes and to establish the capability of the supplier for two-party contractual purposes. The "9001" system deals specifically with quality assurance in the design/development, production, installation and servicing of processes and products. Its purpose is to ensure a supplier's capability to meet the quality expectations of its customers.

The Engine and Drive Train Lubricants (EDTL) Section obtained ISO 9001 registration in December 1993. This is the first ISO 9001 registration for Imperial Oil Products Division. The scope includes the design and development of passenger car, heavy duty, natural gas and two-cycle engine oils, automotive gear oils, tractor hydraulic fluids, automatic transmission fluids and power steering fluids. SRC ISO registration plans for 1994 include Industrial and Base Oils, Engine Test Lab, Analytical and the Customer Support Lab with further expansion in 1995.

APPENDICES

PATENT #	DATE	AUTHOR(S)	TITLE
<u>1992 PATENTS NOT PREVIOUSLY REPORTED</u>			
(1) (US) 5,158,670	92 10 27	I.A. Cody	Hydrocarbon Catalytic Cracking Utilizing a Precoked Catalyst
(2) (US) 5,167,847	92 12 01	C. Olavesen K.D. Butler	Process for Producing Transformer Oil From a Hydrocracked Stock
(3) (US) 5,167,773	92 12 01	A. Welmers	Distillation Tower and Side Stream Stripper Therefor
(4) (US) 5,173,191	92 12 22	L.E. Black	Interfacially Polymerized Membranes for the Reverse Osmosis Separations of Organic Solvent Solutions
(5) (US) 5,169,530	92 12 08	M.M. Hafez	Hollow Fiber Module Using Fluid Flow Control Baffles
<u>1993 PATENTS</u>			
(1) (US) 5,181,998	93 01 26	W.J. Murphy D.H. Shaw	Upgrading of Low Value Hydrocarbons Using a Hydrogen Donor and Microwave Radiation
(2) (US) 5,182,248	93 01 26	I.A. Cody D.H. Dumfries A.H. Neal (ERDL) K.L. Riley (ERDL)	High Porosity, High Surface Area Isomerization Catalyst
(3) (US) 5,183,789	93 02 02	J.P. Boyle	Ozone Regeneration of Platinum, and Polymetallic Platinum Reforming Catalysts
(4) (US) 5,183,566	93 02 02	M.M. Hafez C.P. Darnell (ERDL)	Hollow Fiber Module Built Up From Concentric Cylinders of Hollow Fibers
(5) (US) 5,186,799	93 02 16	W.J. Murphy	Method for Improving the Activity Maintenance of a Plasma Initiator
(6) (US) 5,200,062	93 04 06	M.A. Poirier J.B. Gilbert	Process for Removing Elemental Sulfur from Fluids

PATENT #	DATE	AUTHOR(S)	TITLE
(7) (US)5,199,978	93 04 06	M.A. Poirier D.W. Kraemer I.D. Campbell J.B. Gilbert R.J. Falkiner	Process for Removing Elemental Sulfur from Fluids
(8) (US)5,200,382	93 04 06	I.A. Cody M.M. Hafez D.N. Zinkie	Catalyst Comprising Thin Shell of Catalytically Active Material Bonded onto an Inert Core
(9) (US)5,205,915	93 04 27	A. Ravella W.J. Murphy	Conversion of Methane Using Continuous Microwave Radiation
(10)(US)5,205,912	93 04 27	W.J. Murphy	Conversion of Methane Using Pulsed Microwave Radiation
(11)(US)5,212,128	93 05 18	B.U. Achia J.J. Schorfheide (ERDL)	Method for Recovering or Maintaining the Activity of Hydroisomerization Catalysts
(12)(US)5,220,106	93 06 15	D.R. Boate M.J. Zaworotko (U. of St. Mary's)	Organic Non-Quaternary Clathrate Salts for Petroleum Separation
(13)(US)5,221,465	93 06 22	J.P. Boyle W.C. Baird (ERDL) G.A. Swan III (ERDL)	High Activity, High Yield Tin Modified Platinum-Iridium Catalysts, and Reforming Process Utilizing Such Catalysts
(14)(US)5,221,460	93 06 22	A.R. Dekraker	Continuous Auto-refrigerative Dewaxing Crystallization Using a Centrifuge
(15)(US)5,234,597	93 09 23	A. Welmers L.E. Black	Solvent Extraction Process Involving Membrane Separation of Extract Phase and/or Intermediate Zone Phase with Pseudo Extract/Pseudo Raffinate Recycle, Preferably Employing Interfacially Polymerized Membranes
(16)(US)5,228,977	93 09 23	L.E. Moran W.J. Murphy	Method of Producing Asphalt Having an Increased Penetration and Penetration Index

PATENT #	DATE	AUTHOR(S)	TITLE
(17)(US)5,227,082	93 09 23	L.Z. Pillon A.E. Asselin	Lubricating Oil Having Improved Rust Inhibition and Demulsibility
(18)(US)5,229,021	93 09 23	L.Z. Pillon A.E. Asselin	Wax Isomerate Having a Reduced Pour Point
(19)(US)5,225,094	93 09 23	L.Z. Pillon A.E. Asselin G.A. MacAlpine	Lubricating Oil Having An Average Ring Number of Less than 1.5 per Mole Containing a Succinic Anhydride Amine Rust Inhibitor
(20)(US)5,250,181	93 10 05	I.D. Campbell M.A. Poirier R. J. Falkiner	Process for Removing Elemental Sulfur from Fluids
(21)(US)5,250,081	93 10 05	C.J. May J.J. Habeeb	Smoke Reducing Additive for Two-Cycle Engine Lubricant-Fuel Mixture Comprising the Hofmann Decomposition Products of a Quaternary Ammonium Hydroxide
(22)(US)5,254,795	93 10 19	H.A. Boucher D.L. MacGregor	Removal of 2-Ring Aromatics From Low Boiling Streams Containing Low Concentrations of Same Using Membranes
(23)(US)5,256,297	93 10 26	J.L. Feimer L.T. DesJardine	Multi-Stage Ultrafiltration Process
(24)(US)5,266,175	93 11 30	W.J. Murphy	Conversion of Methane, Carbon Dioxide and Water Using Microwave Radiation
(25)(US)5,269,907	93 12 14	J.P. Boyle W.C. Baird (ERDL) G.A. Swan, III (ERDL)	Process for Reforming at Low Severities with High-Activity, High-Yield, Tin Modified Platinum-Iridium Catalysts
(26)(US)5,269,925	93 12 14	T.E. Broadhurst	Filter Comprising Multiple Layers of Non-Woven Filter Fabric of Decreasing Porosity

FORMAL PROJECT REPORTS - 1993

<u>MONTH</u>	<u>NUMBER</u>	<u>AUTHOR</u>	<u>TITLE</u>
January	IR.1D.93	D. E. Steere H. C. Henry + Research Staff	Sarnia Research Centre - 1992 Review
January	IR.1KT.93	T. E. Broadhurst	Early Efforts to Correlate 600N Dewaxing Performance with Raffinate Feed Quality
January	IR.1BA.93	S. G. Roussis J. W. Fedora	Principles and Methods of Mass Spectrometry Available in Sarnia Research Centre (SRC): A User's Guide
January	IR.1MB.93	S. G. Roussis J. W. Fedora	A Mass Spectrometric Method for the Molecular Characterization of Crude Oils
January	IR.1DC.93	D. W. Blue F. A. Lavji R. W. Scarrow	Water Effluent Quality from Commercial Agency Sites
January	IR.1C-AS.93	M. J. Gale	Athabasca Asphalt Assay
February	IR.1K.93	L. R. Carey D. G. Ryan	Lube Process Annual Report - 1992
February	IR.2KT.93	H. A. Boucher Contr. by: R. W. Saunders D. J. LeClair S. W. Armstrong	The Performance of the Commercial Sarnia Ketone/Oil Membrane Unit 1986-1991, Performance and Characterization of Ketone/Oil Polyimide Membranes and Elements
February	IR.1PK.93	A. Ravella C. A. Swift Contr. by: K. E. Williamson L. E. Moran M. J. Gale	Process Studies on Asphalt Production at Nanticoke
February	IR.1PI.93	D.P. Hakala R.A. Cadwallader	The Lubrication of Industrial Worm Gear Drives

<u>MONTH</u>	<u>NUMBER</u>	<u>AUTHOR</u>	<u>SORTED</u>	<u>TITLE</u>
February	IR.2D.93	D. R. Boate L. E. Black A. G. Blahey H. A. Boucher L. J. Lawlor K. F. Laycock J. M. MacDonald W. J. Murphy M. A. Poirier		Exploratory Research at Sarnia Research Centre - 1992 Review and 1993 Programs
February	IR.3KT.93	D. A. Brandes		Molecular Modelling: Defining a Link between Isoparaffin Structure and Low Temperature Fluidity
February	IR.4KT.93	D. A. Brandes D. N. Zinkie		Exfoliated Alumina as a Novel Support Material for Wax Isomerization
February	IR.5KT.93	R. W. Kapala L. F. Adams		1992 Lube Assays Summary of Predicted Processing Responses
March	IR.2C-AS.93	M. J. Gale		Asphalt Assay - Federated
March	IR.1PH.93	A. G. Blahey K. F. Laycock R. D. Mai P. A. Riddoch J. T. Scales		High Temperature Lubrication Final Report to Energy, Mines and Resources
March	IR.2PH.93	A. G. Alexander C. J. May C. R. Smith		Factors Affecting Pumpability in Heavy Duty Diesel Truck Engines at Low Ambient Temperatures - Part I
March	IR.3PH.93	A. G. Alexander C. J. May C. R. Smith		Factors Affecting Pumpability in Cold Cranking in Heavy Duty Diesel Truck Engines at Low Ambient Temperatures - Part II
March	IR.4PH.93	A. G. Alexander G. A. MacAlpine C. J. May C. R. Smith		Passenger Car and Heavy Truck Cold Cranking Studies - Effects of Oil Viscosity and Vehicle Type

<u>MONTH</u>	<u>NUMBER</u>	<u>AUTHOR</u>	<u>TITLE</u>
March	IR.5PH.93	A. G. Blahey K. F. Laycock	The Effect of Aftermarket Additives on the Performance of Esso Passenger Car Engine Oils
March	IR.1KO.93	M. A. Poirier D. W. Kraemer P. A. Carson G. M. Platten J. S. McKechnie	Development of a Treating Process for the Removal of Elemental Sulphur from Mogas
March	IR.1PG.93	L. Z. Pillon Contr. by: R. S. Polizzotti	Compositional Effects of Base Oils on the Performance of Rust Inhibitors
March	IR.6KT.93	S. J. Alward W. N. Hayter Contr. by: B. J. Whitney M. Q. Tran	Evaluation of Extraction Solvent Selection Techniques
April	IR.7KT.93	D. A. Brandes D. N. Zinkie Contr. by: J. E. Hall	Raffinate Hydroconversion 1992: An Evaluation of the Isomerization Catalyst, Process Variables and Product Quality
April	IR.8KT.93	S. J. Alward W. N. Hayter	Physical Properties of Model Compound Lube Oils
April	IR.2PK.93	B. J. Whitney M. J. Gale L. W. Windsor	Field Performance Site Assessment 1992 - Premium Asphalt Overlay Nanton Alberta Highway 2 - 1989
April	IR.2PG.93	L. Z. Pillon	Foaming of Lube Oils Part 1 - Base Oils
April	IR.3PK.93	B. J. Whitney M. J. Gale	Field Performance Site Assessment 1992 - Sault Ste Marie Engineered Bitumen Overlay - 1989
April	IR.9KT.93	R. W. Kapala L. F. Adams	Lube Assay Procedures

<u>MONTH</u>	<u>NUMBER</u>	<u>AUTHOR</u>	<u>SOURCE</u>	<u>TITLE</u>
April	IR.1OE.93	D. J. LeClair Contr. by: S. W. Armstrong R. J. Basile J. L. Feimer G. D. King B. W. Tobin		B86 Batch Resid Ultrafiltration Unit to Test Ceramic Membranes
May	IR.10KT.93	D. A. Brandes D. R. Boate G. G. Moore J. E. Hall		Process Options and Product Quality Evaluation of an Alternate (4 cSt) Grade Slack Wax Isomerate
May	IR.11KT.93	A. Ravella D. J. Sfalcin		Alternative Catalysts for WISR
May	IR.2PI.93	K. D. Butler M. Serafini W. Nywening		Reformulation of Teresso Steam Turbine/R&O Oils
May	IR.4PK.93	O. Puzic M. J. Gale B. B. Adams		Effect of Used Oil Residue on Asphalt Quality
June	IR.2KO.93	P.A. Carson R.S. Christopher G.L. Archambeault S.A. Thornton Contr. by: L.J. Lawlor B.O. Snider		Demonstration of the Soilwashing/Solvent Extraction Process for the Remediation of Toronto Harbourfront Properties - Part I
June	IR.3KO.93	P.A. Carson R.S. Christopher G.L. Archambeault S.A. Thornton Contr. by: L.J. Lawlor B.O. Snider		Demonstration of the Soilwashing/Solvent Extraction Process for the Remediation of Toronto Harbourfront Properties - Part II

<u>MONTH</u>	<u>NUMBER</u>	<u>AUTHOR</u>	<u>TITLE</u>
June	IR.2OE.93	S. W. Armstrong T. Day J. L. Feimer	Operating Manual for M49 Resid Ultrafiltration (RUF) Test Unit
June	IR.2MB.93	S. G. Roussis J. W. Fedora W. P. Fitzgerald	Gas Chromatographic Distillation/Mass Spectrometry (GCD/MS): Report on its Feasibility to Determine the True Boiling Point (TBP) Curve of Crude Oils
July	IR.1KH.93	A. Uppal L. L. Schinkel	Sulfuric Acid Alkylation - Part 1 Feedstock Effects
July	IR.2KH.93	A. Uppal L. L. Schinkel	Sulfuric Acid Alkylation - Part 2 Appendices to Report IR.1KH.93
July	IR.6PH.93	H. E. Henderson	IOL Basestock Quality Outlook - A Discussion Paper
July	IR.12KT.93	D. J. Bristow A. R. Lindsay	Improvements in Propane Dewaxing Pilot Facilities (1989-1992)
July	IR.1SU.93	D. J. Bristow L. J. Evers J. A. Cappelli D. F. Woolford	Hondo Deasphalting Studies and Generalized Correlations
August	IR.1BW.93	J. L. Feimer W. E. Winter T. R. Halbert T. S. Lin S. W. Armstrong T. M. Day R. L. Eckes R. J. Basile	Resid Ultrafiltration Using Ceramic Membranes Report No. 1 M49-1 and M49-2 Strathcona Plant Test Results
August	IR.1PD.93	M. A. Poirier J. S. McKechnie	Exploration into Methods for Improving Diesel Ignition Quality - Cooperative Research Program with York University

<u>MONTH</u>	<u>NUMBER</u>	<u>AUTHOR</u>	<u>TITLE</u>
August	ER.14PH.93	A.G. Alexander H.E. Henderson (PRD) I.R. Taylor (Abingdon)	Driveline Lubricant Approval Database Report
August	IR.1PC.93	M. A. Poirier J.S. McKechnie	Development of the Mini-TCMAH Treating Technology for Removal of Elemental Sulphur from Kamloops Gasoline
August	IR.5PK.93	B. J. Whitney M. J. Gale L. W. Windsor	Field Performance Site Assessment 1992 Engineered Bitumen 352 Overlay Edmonton Alberta Secondary Hwy 794 - 1989
August	IR.6PK.93	B. J. Whitney M. J. Gale L. W. Windsor	Field Performance Site Assessment 1992 Engineered Bitumen 352 Overlay Craik Saskatchewan Hwy 11 - 1989
August	IR.13KT.93	A. Ravella D. J. Sfalcin	Catalytic Dewaxing with UOP Catalyst
August	IR.14KT.93	H. A. Boucher J. B. Warwick D. J. LeClair	Membrane Deasphalting of Cold Lake Diluent/Bitumen Blends Using Polymeric Membranes
August	IR.2BW-X.93	G. K. Larish D. de la Cruz Contr. by: I. Shelby	NMP/Oil Separation by Membranes, Membrane & Element Fabrication - Part A: Materials Specification
August	IR.3BW-X.93	G. K. Larish H. Nguyen D. de la Cruz Contr. by: J. Opydke M. Snow M. E. Ellenor R. W. Saunders R. C. Bedard	NMP/Oil Separation by Membranes, Membrane & Element Fabrication - Part B: Membrane Production

<u>MONTH</u>	<u>NUMBER</u>	<u>AUTHOR</u>	<u>TITLE</u>
August	IR.4BW-X.93	G. K. Larish D. de la Cruz Contr. by: C. Falcone I. Shelby R. C. Bedard	NMP/Oil Separation by Membranes, Membrane & Element Fabrication - Part A: Materials Specification
September	IR.15KT.93	D. R. Boate G. G. Moore	Port Jerome Severe Hydrofining Study for Extraction Debottlenecking
October	IR.16KT.93	L. F. Adams R. W. Kapala	Lube Assays: Predicted Extraction Response of Distillates from Various Crudes
November	IR.2PD.93	M. A. Poirier	Review on Cetane Improvers
November	IR.3C-AS.93	M. J. Gale	Asphalt Assay - Mandji Crude
November	IR.4C-AS.93	M. J. Gale	Asphalt Assay - Athabasca
November	IR.3MB.93	S. G. Roussis J. W. Fedora W. P. Fitzgerald	Determination of Sulphur Type in Petroleum Samples by Mass Spectrometry
November	IR.1KF.93	L. L. Schinkel R. W. Scales Contr. by: B. Lasko (ERE)	Benicia Clean Fuels Project
December	IR.3PG.93	L. Z. Pillon	Foaming of Lube Oils Part 2 - Factors Affecting the Performance of Antifoaming Agents

PRESENTATIONS AND PUBLICATIONS - 1993

1. Simulated Distillation of Petroleum Fractions by Capillary Gas Chromatography.
Workman, D.S., Noel, F., Watt, M.R., published in the Journal of Chromatographic Science, 1993, Vol. 31, No. 3, pp. 95-99.
2. Rapid Field Methods for the Quantification of Volatile Aromatics (BTEX) and Total Petroleum Hydrocarbons on Soil.
L.J. Lawlor, E.P. Moreton, P.R. Walsh, presented at the EPA International Symposium on Field Screening Methods for Hazardous Waste and Toxic Chemicals, Las Vegas, Nevada, Feb. 24-26, 1993.
3. Use of a Spreadsheet for Preliminary Reactor Design.
A. Ravella, published in Chemical Engineering Progress, February 1993, page 68.
4. Simulated Distillation of Whole Crudes Using Capillary Column Gas Chromatography,
D.S. Workman, R. Proulx, G.D. Dupre, presented at the Pittsburgh Conference and Exposition on Analytical Chemistry, Chicago, Illinois, Feb. 27-March 4, 1993.
5. Canadian Light Duty Vehicle Emissions Inventories for the Year 2000.
G. Felsky, presented at the SAE International Congress, Detroit, Michigan, March 1, 1993.
6. The Effect of Canadian Gasoline Composition on Exhaust Emissions.
G. Felsky, presented at the SAE International Congress, Detroit, Michigan, March 1, 1993.
7. Synergy Between Additives in Stimulating Diesel Fuel Ignition.
P.Q.E. Clothier, H.O. Pritchard, M.A. Poirier, published in Combustion and Flame, 95, 427-429(1993).
8. Quench Oil Review.
P.J. Kozak, presented at the Can-Eng Continuous Furnace User's Conference, Toronto, Ontario, April 28, 1993.
9. The Future of Canada. Technolog: A Leader or a Follower?
H.C. Henry, presented to the Hugh O'Brien Youth Foundation (HOBY), May 1, 1993.
10. R&D Leveraging Through ICST.
H.C. Henry, presented to the Canadian Chemical Producers Association (CCPA) Research and Development Committee, May 18, 1993.
11. Characterization of Wax Fractions from Lube Oil Basestocks by Thermal Extraction GC/MS.
J.W. Fedora, S.G. Roussis, T.R. Ashe, presented at The 41st ASMS Conference on Mass Spectrometry and Allied Topics, San Francisco, May 30-June 4, 1993.

12. Evaluation of Mass Spectrometric Methods of Determination of Sulfur Types in Petroleum Samples.

J.W. Fedora, S.G. Roussis, W.P. Fitzgerald, presented at The 41st ASMS Conference on Mass Spectrometry and Allied Topics, San Francisco, May 30-June 4, 1993.

13. Measuring Customer Satisfaction.

H.C. Henry, presented at the Canadian Research Management Association (CRMA) Research Practices Workshop, June 15, 1993.

14. Internal Market Economy.

H.C. Henry, presented at the Canadian Research Management Association (CRMA) Research Practices Workshop, June 15, 1993.

15. Optimization of the ASTM D892 Foam Test.

L. Pillon, A.E. Asselin, presented at the ASTM Meeting, Boston Mass., June 29, 1993.

16. Technologists: A Front Line Look at ISO 9000.

C. A. Cardy, published in CIC Chemical News, September, 1993.

17. Quality in Industrial R&D.

H.C. Henry, presented at the 43rd Canadian Chemical Engineering Conference, October 5, 1993.

18. The Impact of Diesel Fuel Quality and Engine Technology on Polyaromatic Hydrocarbon Emissions.

D.E. Steere, presented at the Canadian Chemical Engineering Conference, Ottawa, October 6, 1993.

19. The Performance of Carborane Modified Siloxane Phases Versus Polymethylsiloxane Phases for High Temperature Capillary Simulated Distillation.

Workman, D.S., presented at ASTM Committee D-2 on Petroleum Products and Lubricants, Dallas, Texas, Dec. 6, 1993.

20. Canadian Government Standards Board (C.G.S.B.) Method for Detailed Analysis of Gasoline.

Parr, S.W., Davidson, B.E., presented at ASTM Committee D-2 on Petroleum Products and Lubricants, Study Group for the High Resolution GC Analysis of Gasolines, Dallas, Texas, Dec. 6, 1993.

21. Utilization of LIMS Technology to Facilitate Changes in Business.

Watt, M.R., Bedard, M.A., Watt, S.T., Cain, T.P., published in American Laboratory, December 1993, page 20H-20J.

22. Proposed Revisions to ASTM D-5236 Vacuum Potstill Distillation Procedure.

Hunter, A.J., presented at ASTM Committee D-2 on Petroleum Products and Lubricants, Boston, Mass., December 8, 1993.

RESEARCH EXCELLENCE AWARDS

There are three basic criteria for the Research Excellence Award and Team Excellence Award.

- (i) Excellence in Science and Technology.
- (ii) Excellence in Support of Research Activities and in Support of Research Customers.
- (iii) Excellence in Management, Administration, Systems and Public Relations.

Generally, the Award will recognize achievement in any Research activity or endeavor which promotes discovery, understanding, application, efficiency and productivity within our organization. As well, the Award will recognize efforts at improving our personal skills and involvement in community activities reflecting responsible Corporate citizenship. The Award will recognize short term exceptional effort or long term, consistent effort over a period of time.

Specifically, the Award will recognize creative solutions to problems, development of significant improvements in processes, products, analytical methods, word processing systems, etc., improving efficiency of operations or helping our customers either through technology application or problem solving. In addition, the Award will celebrate the milestones of receiving a post-secondary diploma or degree or receiving several significant patent awards. As the Award matures, other examples will emerge to serve as further guidelines.

1993 Research Excellence Award recipients include:

Dan Sfalcin	For Providing Excellence in Advancing Lube Catalytic Process Research
Rose Smith	Leadership in Laboratory Catalyst Testing
Heather Boucher	Sustained Excellence in the Generation of Innovative Ideas Leading to Patent Memoranda
Frank Linek	For excellence in providing a powerful customer friendly database for the Lubes Hydroprocessing Section
Leo DesJardine Ben McKillican	For excellence in troubleshooting in the development of a novel membrane separation process
Alan Blahey Roger Kilbreath Kim Laycock Rainer Mai Scott McPhail Pam Riddoch Jim Scales	For Excellence in the Execution, Support and Completion of the Development and Validation of Novel Heavy Duty Lubricants for the Low Heat Rejection Heavy Duty Engine

Greg Lawrence Bob Maguire Gary Woods	For Excellence in Blending Engine and Drive Train Lubricants for Research Field Test Programs
Ashok Uppal	For Excellence in the Performance, Completion and Reporting of the Alkylation Pilot Plant Program
Sandra Alward Alberto Ravella	For excellence in providing computer training in EXCEL and Charisma

1. A review of the role of lubricants in the development of the Canadian vehicle industry. Consideration should be given to the contribution of lubricants to the development of the Canadian vehicle industry.
2. The impact of the introduction of the Canadian government's new environmental protection laws on the Canadian oil industry.
3. The impact of the introduction of the Canadian government's new environmental protection laws on the Canadian oil industry.
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21. The impact of the introduction of the Canadian government's new environmental protection laws on the Canadian oil industry.
22. The impact of the introduction of the Canadian government's new environmental protection laws on the Canadian oil industry.

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A. Welmers - E

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 S. A. Thornton
 K. A. Totten
 F. VanderVaart
 E.W. van Kalmthout
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 J. D. Young

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N.M. Bourque	H. M. Murphy	T.L. Ferns	

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J. M. Holbrough

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Mike Keohan

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A. C. Cates
N. Hadj-Bagheri
S. A. Kornelsen
K. L. Sokol
K. C. Teather

PERSONNEL ITEMS

Additions and deletions to career staff at the Sarnia Research Centre (IOL) during 1993 are listed below:

Transfer Ins

J. W. Clamp - Sarnia Refinery
G. C. Lahn - Florham Park (loan in)
G. P. Schleicher - Sarnia Refinery
D. S. Sinclair - Sarnia Refinery
Y. St. Germain - Montreal

New Hires

Z. Gao
J. G. Overend
K. A. Totten

Transfers Out

C. D. Baily - IOL, Montreal
D. A. Brandes - IOL, Calgary
B. J. Cappellazzo - IOL, Toronto
C. A. Michael - IOL, Toronto
L. E. Moran - Marketing, Toronto
G. M. Platten - ECC, Sarnia
L. L. Schinkel - Sarnia Refinery
C. R. Smith - IOL, Toronto
J. R. Sweet - IOL, Toronto

Retirements/Resignations

D. L. Brown
P. C. Carson
M. C. Graham
P. J. Kozak
H. W. Lingley
M. G. Taylor
M. Tran
R. D. Vaillancourt
G. F. Whiteside

